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Technical Report

No. ________

Development of Embedded Acoustic Waveguides for Monitoring Composite Material Processing and Non-Destructive Evaluation

Final Report:
- Phase I - Research Study
- Phase II - Definition of Military and Industrial Areas

December 1995

Contract: DAAE07-93-C-R121
TARDEC Dual-Use Research & Development Project:

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U.S. ARMY TANK-AUTOMOTIVE COMMAND
RESEARCH, DEVELOPMENT & ENGINEERING CENTER
Warren, Michigan 48397-5000

NOTICES

Unclassified:

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188
Exp. Date: Jun 30, 1986

1a. REPORT SECURITY CLASSIFICATION Unclassified	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION / AVAILABILITY OF REPORT
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S)
6a. NAME OF PERFORMING ORGANIZATION Westinghouse Science & Technology Center — 6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION U.S. Army (TARDEC)
6c. ADDRESS (City, State, and ZIP Code) 1310 Beulah Road, Pittsburgh, PA 15235	7b. ADDRESS (City, State, and ZIP Code)
8a. NAME OF FUNDING / SPONSORING ORGANIZATION National Automotive Center — 8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
8c. ADDRESS (City, State, and ZIP Code) Warren, Michigan 48397-5000	10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO. / PROJECT NO. / TASK NO. / WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)

Embedded Acoustic Waveguides for Monitoring Composite Material Processing & N.D.E.

12. PERSONAL AUTHOR(S) Ronald T. Harrold

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Final	FROM 93 Oct TO 95 Sept	95 Dec	131

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	Acoustic Waveguides, Sensors, Liquid Composite Molding, R.T.M. Viscosity, Gelatinization, Modulus, Resin, Composite Material Mold Filling, Bondline, N.D.E., Structural Health Monitoring

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Objectives of this dual-use project were to develop acoustic waveguide (AWG) sensors for monitoring the processing conditions (viscosity, gelatinization, hardening) during laboratory liquid composite molding, and to use the embedded AWG for structural health monitoring of the composite parts. Benefits to both the U.S. Army and the U.S. automotive industry are better quality and lower cost parts, and lifetime structural health monitoring. AWG sensors and programmable instrumentation were developed to yield a degree of cure curve for liquid molded composite parts; and sense mold filling and resin gas content. In the area of NDE development it was determined that AWG can function as strain sensors within composite materials and also yield information on the strength of bondlines. A video has been made which shows AWG cure monitoring of a composite part. Four scientific papers have been written and published in regard to the work performed under this contract. It is recommended that the techniques developed on this project be transitioned to actual factory-type of resin transfer molding machines.

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT ☐ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION
22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code) — 22c. OFFICE SYMBOL

DD FORM 1473, 84 MAR

83 APR edition may be used until exhausted.
All other editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE
Unclassified

ACKNOWLEDGEMENTS

The author is pleased to acknowledge the help of L. E. Ottenberg who assembled all the test apparatus and carried out experimental work and E. S. Diaz for valuable consultation in regard to strain measurement and bondline shear strength tests, and also Z. N. Sanjana for consultation on composite materials and molding procedures. Major Richard Brynsvold provided valuable help in the area of liquid composite molding.

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ABSTRACT

Objectives of this dual-use project were to develop acoustic waveguide (AWG) sensors for monitoring the processing conditions (viscosity, gelatinization, hardening) during laboratory liquid composite molding, and to use the embedded AWG for structural health monitoring of the composite parts. Benefits to both the U.S. Army and the U.S. automotive industry are better quality and lower cost parts, and lifetime structural health monitoring. AWG sensors and programmable instrumentation were developed to yield a degree of cure curve for liquid molded composite parts; and sense mold filling and resin gas content. In the area of NDE development it was determined that AWG can function as strain sensors within composite materials and also yield information on the strength of bondlines. A video has been made which shows AWG cure monitoring of a composite part. Four scientific papers have been written and published in regard to the work performed under this contract. It is recommended that the techniques developed on this project be transitioned to actual factory-type of resin transfer molding machines.

1.0 INTRODUCTION

This final technical report prepared by Westinghouse Science & Technology Center, for the U.S. Army Tank Automotive Command (TACOM) under Contract DAAE07-93-C-R121, describes the laboratory development of embedded acoustic waveguides (AWG) sensors for monitoring the processing conditions (resin viscosity, gelatinization, hardening) during liquid composite molding and the use of embedded AWG for lifetime structural health monitoring of composite materials. This dual-use project which was funded by the National Automotive Center, was planned for application to composite parts suitable for both military and civilian automotive vehicles. Monitoring is needed during liquid composite molding because there is no broadly accepted method available for routinely measuring the processing conditions of resin viscosity, gelatinization and hardening. Consequently, the transition of this AWG technology to factory-size liquid composite molds should benefit both the U.S. Army and the U.S. Automotive Industry by the manufacture of better quality and lower cost parts, and also by the capability for lifetime structural health monitoring.

2.0 OBJECTIVES:

Overall Objective of Program:

Investigate Acoustic Waveguide Cure Monitoring to Improve the Processing of Composite Materials in Order to Manufacture Better Quality and Lower Cost Parts

Application Area: Liquid Composite Molding:

Addressing: 1. Mold Filling and Acoustic Waveguide Cure Monitoring

2. Non-Destructive Evaluation and Structural Health Monitoring

Detailed Objectives:

1. Mold Filling and Acoustic Waveguide Cure Monitoring:

- Design and construct small liquid composite molds for complex parts (right-angled bends)
- Develop AWG cure monitoring so that the cure curves define the curing stages of viscosity, gelatinization, hardening.
- Apply AWG sensors to estimate the degree of mold filling.
- Apply AWG sensors to sense the gas content and voids in the resin during curing.

- Address the theoretical aspects of resin viscosity, gelatinization and hardening.
- Design and apply AWG cure monitoring to a generic part suitable for both military and commercial vehicles.
- Design and construct acoustic meter for cure monitoring.
- Transfer AWG cure monitoring techniques and instrumentation to TARDEC.

2. Non-Destructive Evaluation and Structural Health Monitoring:

- Develop embedded AWG sensors for measuring bondline integrity.
- Develop embedded AWG sensors for measuring strain inside composite materials.
- Investigate embedded AWG sensors for measuring residual strain.
- Recommend best use of embedded AWG for lifetime structural health monitoring (battle damage assessment).

3.0 CONCLUSIONS

During the course of this acoustic waveguide contract, we have carried out extensive experimentation using model liquid composite molds. A large amount of data have been obtained and we have gained considerable knowledge relating to the AWG sensing of the curing process within composites in liquid molds, and also AWG N.D.E., quality and structural health monitoring. These accomplishments and AWG applications are summarized below:

AWG Sensing of Processing Conditions Within Liquid Composite Molds:

It has been demonstrated that:

- AWG sensors will work around 90° bends in composite parts.
- AWG sensors can provide approximate values for resin viscosity prior to gelation.
- AWG sensors can pinpoint gelation phase of resin.
- AWG sensors can provide an approximate value for the resin modulus during hardening after gelation.
- AWG sensors can provide a measure of mold filling.
- AWG sensors can yield information on the resin gas (bubble) content after pouring of resin.
- Apart from AWG attenuation measurements, wave velocity readings can sometimes provide a cure curve.

AWG Sensors for N.D.E. Quality and Lifetime Structural Health Monitoring:

It has been demonstrated that:

- AWG sensors can monitor real time compressive and tensile strain inside molded composite parts.
- AWG sensors can yield information on the residual strain and modulus of molded composite parts.
- AWG sensors can provide some information on bondline integrity.
- AWG sensors arrays have the potential to monitor composite damage and identify its type and location.
- AWG sensor arrays have the potential to monitor the void content of molded composite parts.

4.0 RECOMMENDATIONS:

Having now established a firm basis and a better understanding of AWG composite process monitoring and N.D.E. applications, the need now is to transition this technology to an actual resin transfer molding machine and practical size composite parts. The outline for a follow-on transition program, the issues involved and tasks is given in Appendix A, and is entitled:

Transition of AWG Laboratory Cure Monitoring and N.D.E. Techniques to Actual R.T.M. Machine and Practical Size Parts

5.0 DISCUSSION

5.1 Background to the Acoustic Waveguide Development Program

Due to their high strength and low weight, composite materials are attractive for the construction of tanks, aircraft and automobiles, all of which require the optimum use of fuel. Factors restricting the widespread use of composite materials are high material and manufacturing costs and the lack of a reliable data base of strength and fatigue life which designers have for metal, for example. One way of reducing costs and obtaining better quality composite parts is to improve process monitoring, so that as parts are manufactured the curing conditions inside the bulk of the parts are known and understood, and processing conditions can be changed to improve quality. Another way is to have sensors embedded within composite parts so that during their lifetime measurements can be made of material strength, and fatigue damage assessed.

Embedded acoustic waveguide (AWG) sensors, such as, 10 to 20 mil diameter Nichrome wires, are simple sensors which do not degrade the strength of composite parts, and can yield information on processing conditions during manufacture and structural strength during their lifetime.

The purpose of this dual-use program on embedded AWG was to apply these sensors to laboratory liquid composite molding; understand the phenomena involved and determine their sensitivity and performance in the areas of mold filling, gas and void sensing, and cure monitoring (viscosity, gelatinization, hardening). In addition, in the area of N.D.E., emphasis was placed on measuring bondline integrity and assessing internal strain. To achieve the program objectives, two phases were carried out as listed below, and finally the technology transferred to TARDEC and suggestions made for a follow-on program in order to transition this embedded AWG technology to actual full-size liquid composite molding machines.

PHASE I - Research Study

PHASE II - Definition of Military and Industrial Areas

5.1.1 The Phase I Tasks are Listed Below:

C.2 Phase 1: - Research Study:

Task:

C.2.1 Design and Construct Representative Liquid Mold

C.2.2 Study AWG Cure Monitoring of Complex Part

C.2.3 Investigate Mechanical Strength of Parts with AWG

C.2.4 Study Residual Strain in Right Angle Bend with AWG

C.2.5 Apply Cure Monitoring using Composite Part as AWG

C.2.6 Study Incomplete, Rapid Mold Filling, AWG Void Sensing

C.2.7 Study AWG Sensing of Bondline Integrity

C.2.8 Conduct Mechanical Tests of Bondline

C.2.9 Conduct Modulus Tests on Specimen as Required

C.2.10 Make NDE Evaluation; Structural Health Recommendations; Economics

5.1.2 The Phase II Tasks are Listed Below:

C.3 Phase II: Definition of Military and Industrial Areas

Task

C.3.1 Design Generic Part with Embedded AWG

C.3.2 Design and Construct Mold for Generic Part

C.3.3 Optimize Generic Part, AWG Placement, Cure Curve

C.3.4 Exploratory AWG, NDE Experiments to Enhance Sensing Voids; Residual/Real Time Strain; Bondline; Mold Fill

C.3.5 Mechanical/Modulus and Associated Tests as Necessary

C.3.6 Address Theoretical Aspects of Viscosity/Modulus; Strain; Bondline Integrity and Void Sensing

C.3.7 Design and Construct/Calibrate Sonic Meter Sensing System for AWG Cure Monitoring

C.3.8 Transition Laboratory AWG Cure Monitoring System to Practical Demonstration at TARDEC

5.2 Curing of Materials:

Generally, polymeric composite materials, whether cured at room temperature within a mold, or cured in a heated press or an autoclave, proceed through three district stages before final cure. These stages are, (1) large increase in viscosity, (2), gelatinization (transition from a liquid state to a rubbery gel) and (3), hardening or increasing modulus to final cure. A further stage related to cure occurs just after pouring of resin into a mold, when gas within the resin or trapped gas is dispersing within or exiting from the resin. For processing control purposes, the stages of mold filling, gas dispersion and increasing viscosity are most important and require careful monitoring, because there is still time to change pressure or temperature, for example, and optimize the cure cycle.

The three stages of curing are well defined even if the resin cures in a very short time, minutes, or very long time, days. For example, acoustic waveguide cure curves for a fast cure resin and a slow cure concrete are shown in Figures 5-1 and 5-2 respectively. It can be seen that the fast cure resin reaches gelatinization in about 12 minutes, while for concrete the time is about 3 days. Final cure for the concrete occurs in about 70 days and surprisingly, final cure for the resin occurs in about 2 to 3 days.

5.3 Acoustic Waveguides and How They Work:

Five hundred years ago Leonardo da Vinci put one end of a long wooden tube into the ocean, the other end to his ear, and listened to the approach of distant ships. In this case the sounds from ships travelled through the seawater to the wooden tube, and then through the wooden tube (acting as an acoustic waveguide (AWG) to the ear. This may be the first documented experiment using an AWG. Another simple example is a string stretched between two cans for voice transmission. The string is acting as an acoustic waveguide and the cans as transmitters and receivers. The degree of acoustic wave transmission within an AWG depends upon whether a material is hard or soft, and on its density and acoustic wave velocity. Hard materials with a low Poisson's ratio (ratio of change in diameter to change in length of a rod under tension) transmit best, while soft materials tend to damp out wave transmission. Larger diameter rods transmit larger signal values, but even small (10 to 20 mil) diameter rods and wires can be used with useful signal levels from a frequency of ~1 kHz to ~400 kHz. An advantage of AWG rods is that they can be used over a wide frequency range, and diameter is not as critical as in the case of rectangular radar waveguides which do not work unless they are precisely dimensioned to a size of one half wavelength.

The basic concept[1] of AWG cure monitoring is that the amount of sound transmitted through an embedded AWG is closely related to the difference between the acoustic impedance of the AWG and the acoustic impedance of, and the pressure exerted by, the host material which surrounds the AWG. The acoustic impedance of the medium which surrounds the AWG depends upon the density of the host material times the velocity of sound through it. Thus, any physical or chemical changes which affect the density of the host material or velocity of sound through it will alter the acoustic impedance and change the amount of sound transmitted through the AWG. Since the density and velocity of sound through a host material are affected by temperature, stress, strain and impact on the host material, all these things can be monitored by means of the AWG. The transit time of the sound wave through the AWG is also affected by these factors and can also be used to monitor changes that occur in the host material.

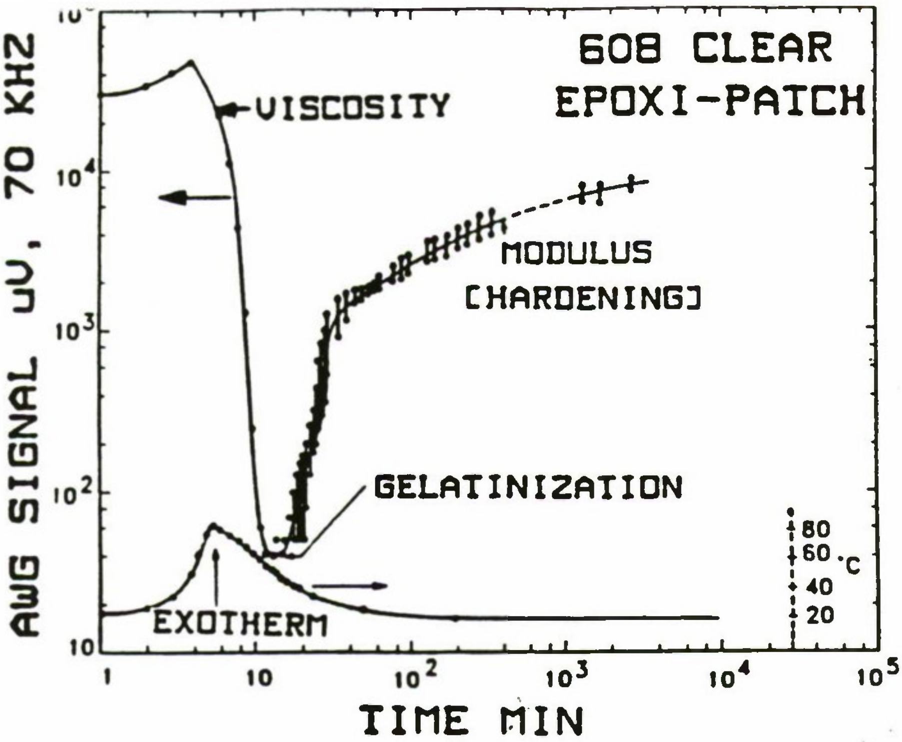


Figure 5-1. Acoustic waveguide cure curve for fast cure resin

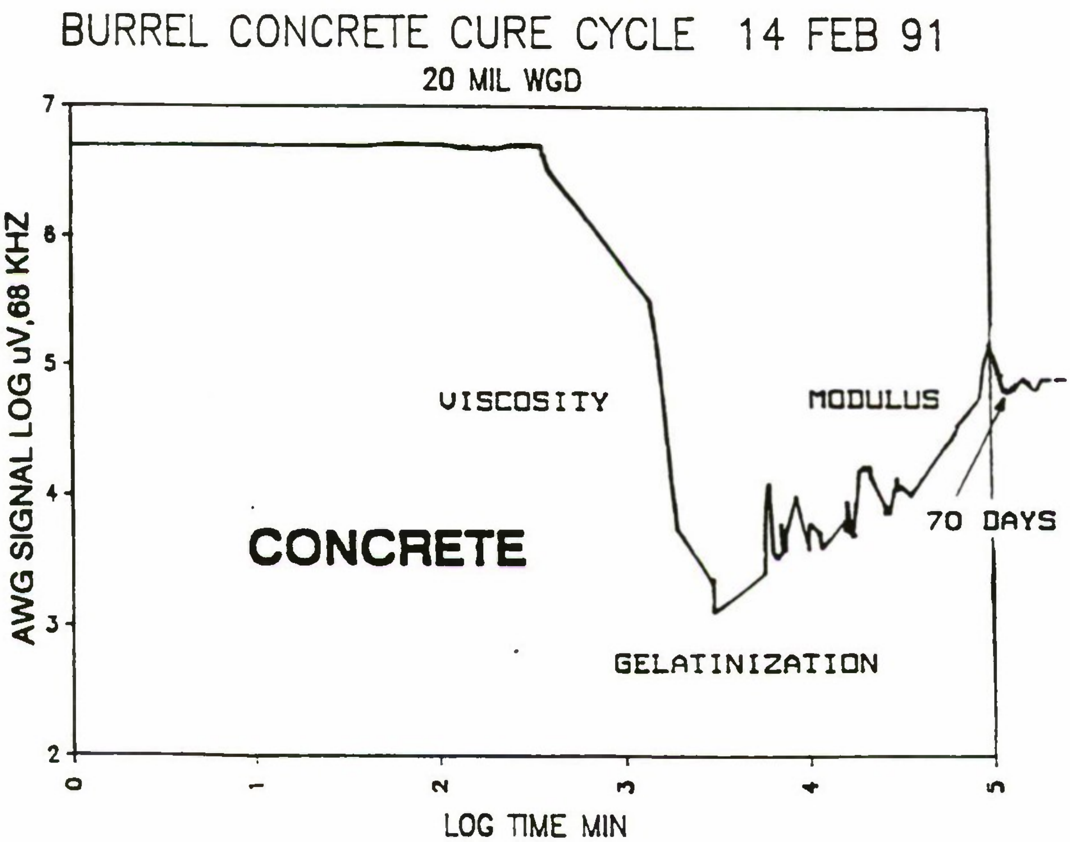


Figure 5-2. Acoustic waveguide cure curve for slow cure concrete

5.4 PHASE I - Accomplishments and Results:

The significant accomplishments and results of the Phase I - Research Study are outlined and described in the following:

5.4.1 Resin Part with Two Right-Angled Bends:

In order to reduce time and cost, and also to obtain practical data from a large number of experiments, a simple mold into which room temperature resins could be poured was designed, constructed and used. One version of this mold is shown in Figure 5-3 where it can be seen that the small molded parts which were produced have two right-angled bends. The right-angled bends were planned so as to determine that AWG would function for cure monitoring and N.D.E. purposes when complex shaped molded composite parts are required in the future for military and civilian vehicles.

Another purpose of this part with two right-angled bends was ease of use for measurements of internal strain later in the program. As can be seen in Figure 5-3 the basic instrumentation required for AWG monitoring of resin cure and N.D.E., is a pulse function generator; acoustic transducers functioning as acoustic transmitters and receivers in the 60 to 80 kHz signal range; a high pass filter and oscilloscope, and a T.V. camera, V.C.R. and T.V. monitor. Basically, electrical pulses at the rate of 150 per second in bursts of 32 cycles around 60 to 80 kHz (the resonant frequency of the system), are fed to the acoustic transmitter which is bonded to one end of AWG within the mold. The acoustic transmitter changes the electrical pulses to acoustic pulses which are transmitted through the AWG within the mold. Depending upon the state of the curing resin within the mold, the magnitude and wave velocity of these pulses reduce as they travel through the AWG. At the termination of the AWG the acoustic receiver converts these acoustic pulses back to electrical pulses which are displayed on an oscilloscope. It is the peak magnitude of the received pulse which is used to plot a resin cure curve versus time. A 30 kHz, high pass filter is used to filter out background noises and a video recording of the oscilloscope screen is made if necessary so as to accurately measure rapidly changing signal levels or other transient events.

Throughout this program a clear resin was used so that defects and bubbles could be seen inside molded parts and optical strain measurements could also be made. For example, the resin used was Shell 815 (a bisphenol - A - epoxy) which has low viscosity and flows readily through molds with glass fibers inside. The hardener used with this resin was from Huntsman Corporation and called Jeffermine T-403 and also an accelerator 399 from Huntsman Corporation was used. A typical mixture would be 20 gms of 815, 8.8 gms of T-403 and 2 gms of 399. The weight ratios being 65%, 28.5% and 6.5% respectively, and the room temperature curing time was around 400 minutes.

In Figure 5-4 a molded part of Shell 815 resin is shown with an embedded 10 mil diameter Nichrome AWG, while in Figure 5-5 another part is shown with four plastic inserts to hold the AWG in position. During the molding of composite parts the AWG is supported by the strengthening glass fibers that are used.

5.4.2 Mold for Resin Part

In Figure 5-6, the aluminum mold for the right-angled resin parts is shown. Prior to mold filling (resin poured into one end of mold) the inside of the mold is coated with mold release. The

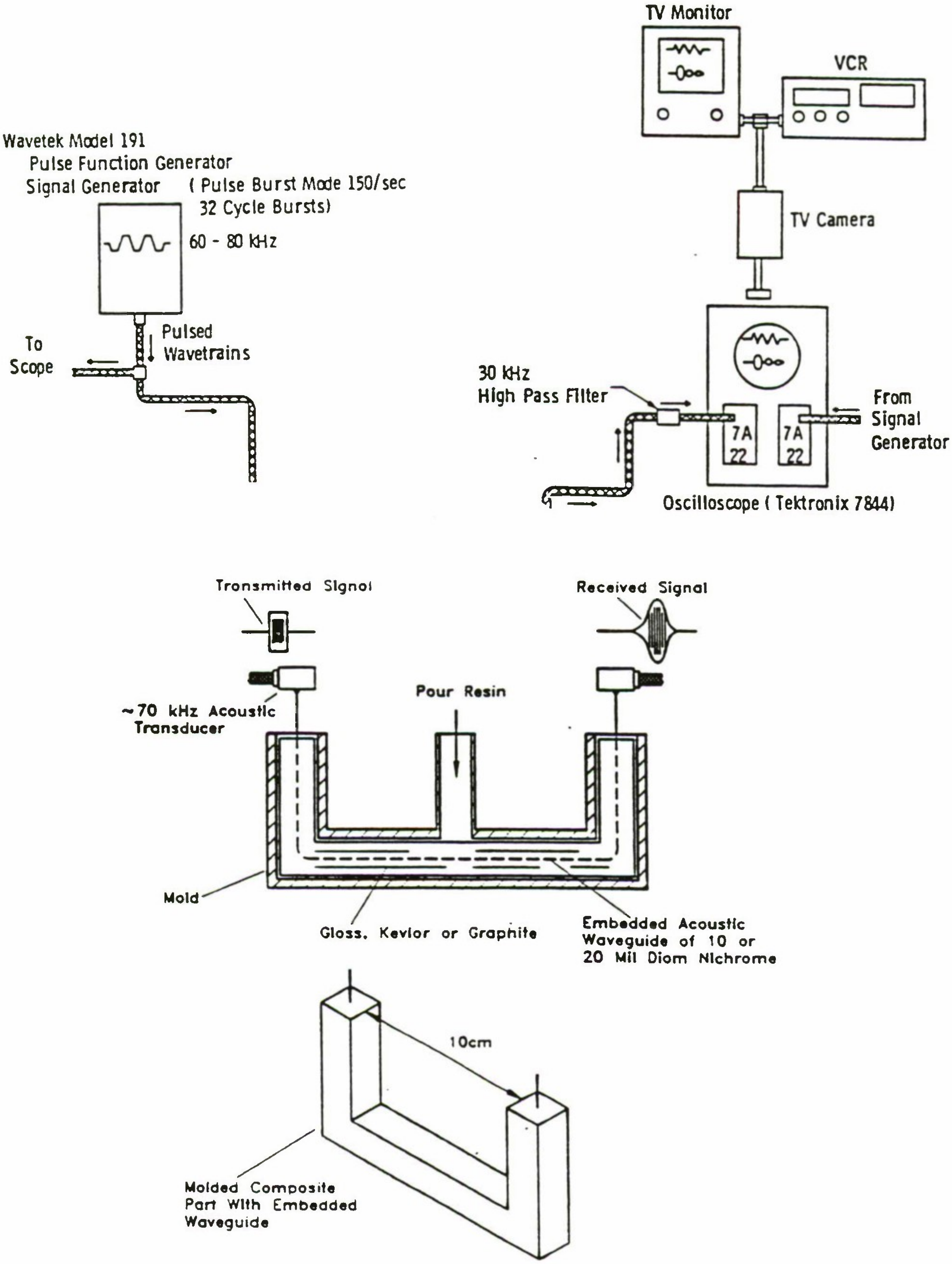


Figure 5-3. Scheme for acoustic waveguide cure monitoring of molded composite part

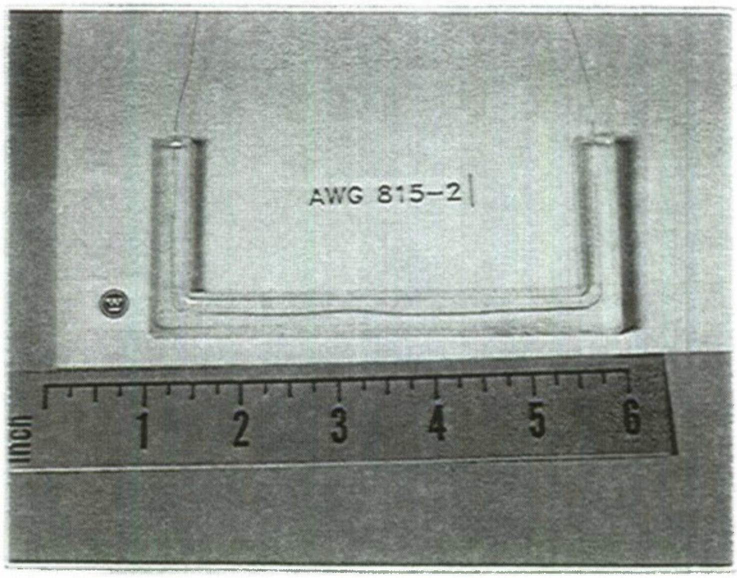


Figure 5-4. Molded part of Shell 815 resin with two right angle bends and an embedded acoustic waveguide of 10 mil diameter Nichrome

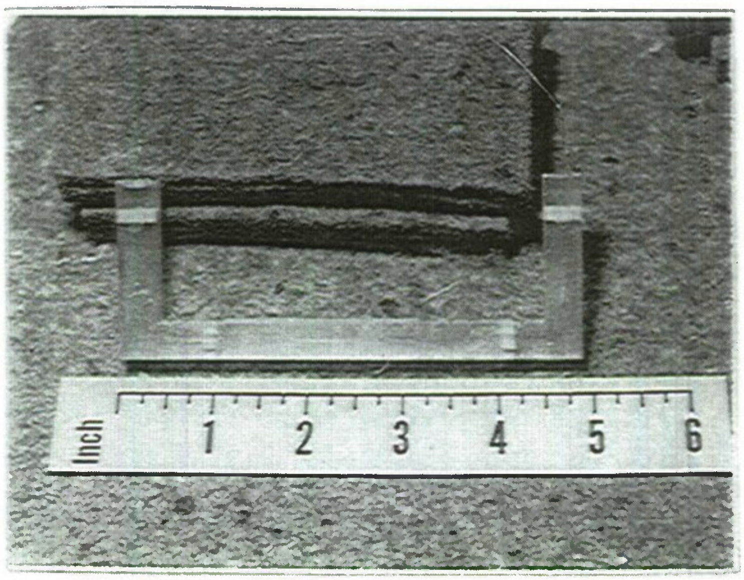
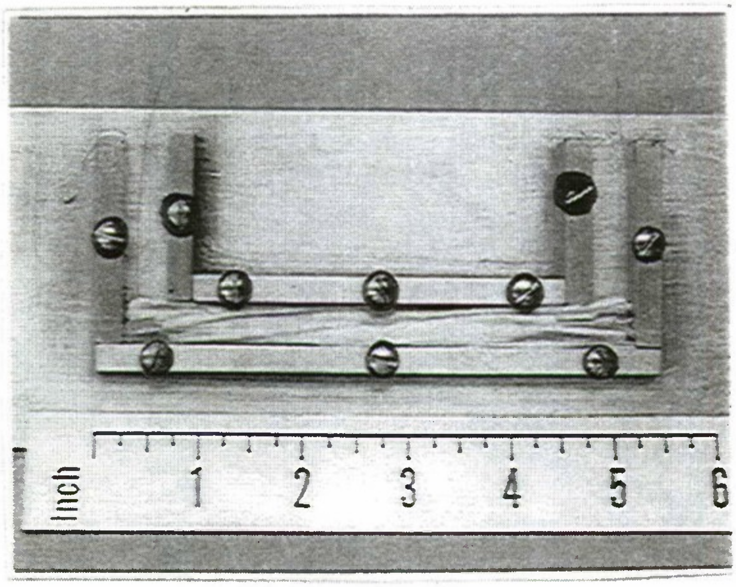


Figure 5-5. Right-angled part of Shell 815 resin with four plastic inserts for centralizing the AWG sensor (from prototype mold)

Nichrome AWG



Mold with transparent Plexiglas face allows viewing of "E" glass fibers and Nichrome AWG.

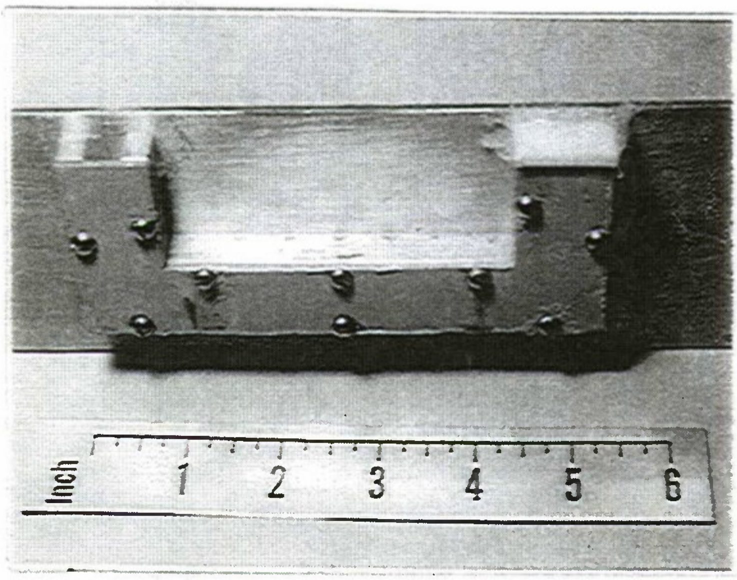


Figure 5-6. Prototype mold for right-angled resin parts

front of the mold screws into position and can be removed for extracting the molded part following complete cure. The AWG is held in position via Teflon end pieces as seen in the right-hand side of the mold Figure 5-6 or by plastic inserts, Figure 5-5. An overall picture of the mold and cure monitoring instrumentation is shown in Figure 5-7. The cure curve shown on the T.V. screen was plotted from the magnitude of the received signal levels obtained from the oscilloscope screen.

5.4.3 Acoustic Waveguide Cure Curves for Resin Part

Acoustic waveguide cure curves for the small resin parts with two right-angled bends are shown in Figure 5-8. These resin parts were made in the prototype mold, Figure 5-6, and infra-red heat was applied to speed up the curing process. Although data from both the AWG transmitted signal levels and acoustic wave velocity within the AWG are plotted, the AWG signal levels represent the cure curve. It can be seen that just after time zero, when the resin has been poured into the mold, the AWG signal rises slightly for a few minutes and this may be due to trapped gas dispersing within the resin. Next, the AWG signal falls rapidly due to the large increase in resin viscosity as polymerization occurs. The signal level falls about 500 times in value (logarithmic scale) to reach a plateau at gelatinization (the transition from a liquid state to a rubbery gel). This gelatinization period is followed by a rising signal level as the resin hardens and the modulus increases until final cure. The sudden rise in signal level at final cure is due to the removal of the heat from the infra-red lamp. It can be seen that the AWG velocity data also forms a cure curve, Figure 5-8, but this can be difficult to measure and does not always give sufficient information for a cure curve. The reason why the velocity data relates to the curing process is because the velocity of acoustic waves within the AWG depends upon the acoustic impedance (density times wave velocity) of the resin which changes with viscosity and modulus.

5.4.4 Acoustic Waveguide Cure Curves for Composite Part

A composite part was made using 26.6% by weight of "S" glass fibers. These glass fibers were bundled and placed within the mold prior to filling with Shell 815 resin. The low viscosity resin flowed readily through the fibers to fill the mold. The AWG cure curve for this composite part is shown in Figure 5-9 and this curve is similar to the cure curves for resin only, Figure 5-8. Differences are a larger rise in AWG signal level just after mold filling which could indicate more gas dispersion within the resin due to the glass fibers causing gas entrapment; and less time in the gelatinization region. Also, the AWG wave velocity data does not give a good measure of the curing cycle. In Figure 5-10, a picture shows a composite part made from Shell 815 clear resin and containing 33% by weight of "S" glass fibers.

5.4.5 Cure Curve Using Part as Acoustic Waveguide

A major advantage of using embedded AWG sensors for cure monitoring is the ability to monitor processing conditions inside complex-shaped parts and actually inside of molds. An alternative approach, where long slender-shaped parts are involved, may be to use AWG stubs at the entry and exit to a mold, one to transmit signals and one to receive signals, and utilize the curing part as an acoustic waveguide. This approach was tried and the resulting cure curves are shown in Figure 5-11. It can be seen that gelatinization can be identified and the hardening or increasing modulus phase of curing is clearly measured and defined. However, there is no indication of the large increase in resin viscosity which occurs during the first seventy minutes of the curing cycle. This is to be expected because as the part in acting as an AWG then very little

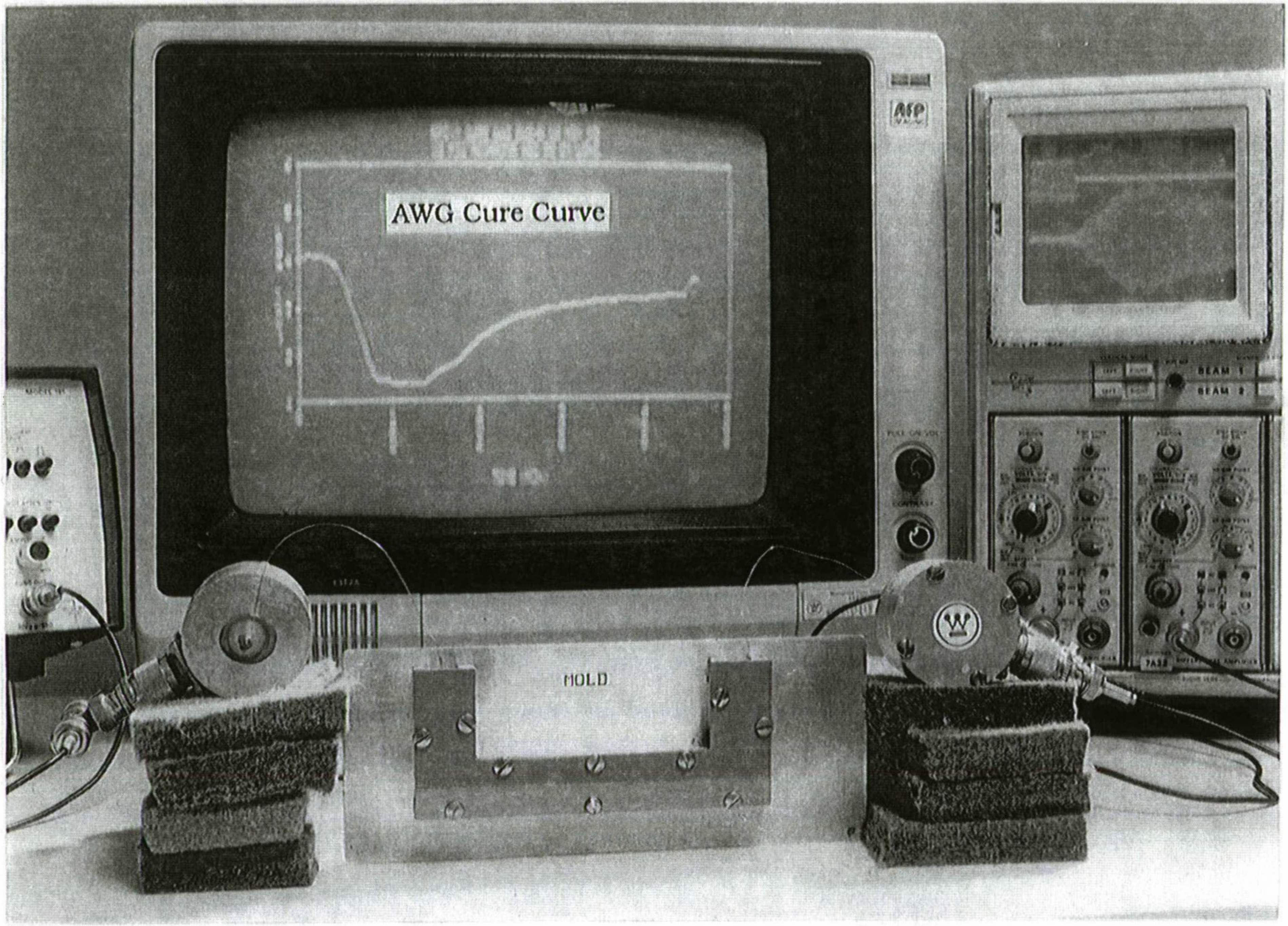
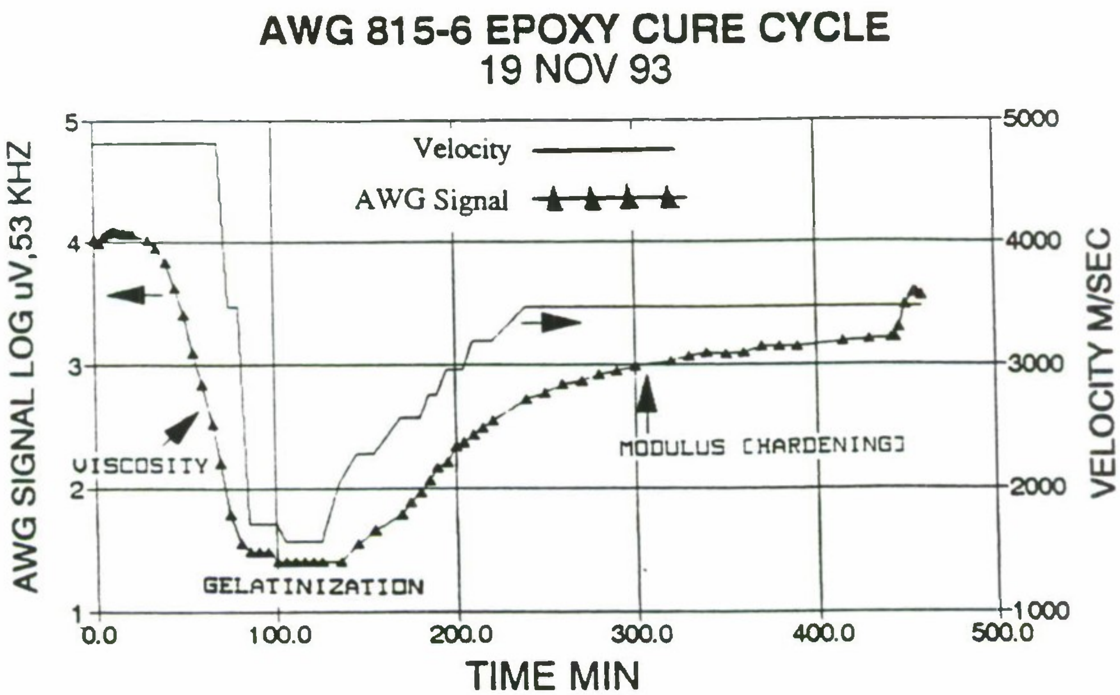


Figure 5-7. **AWG Cure Monitoring**



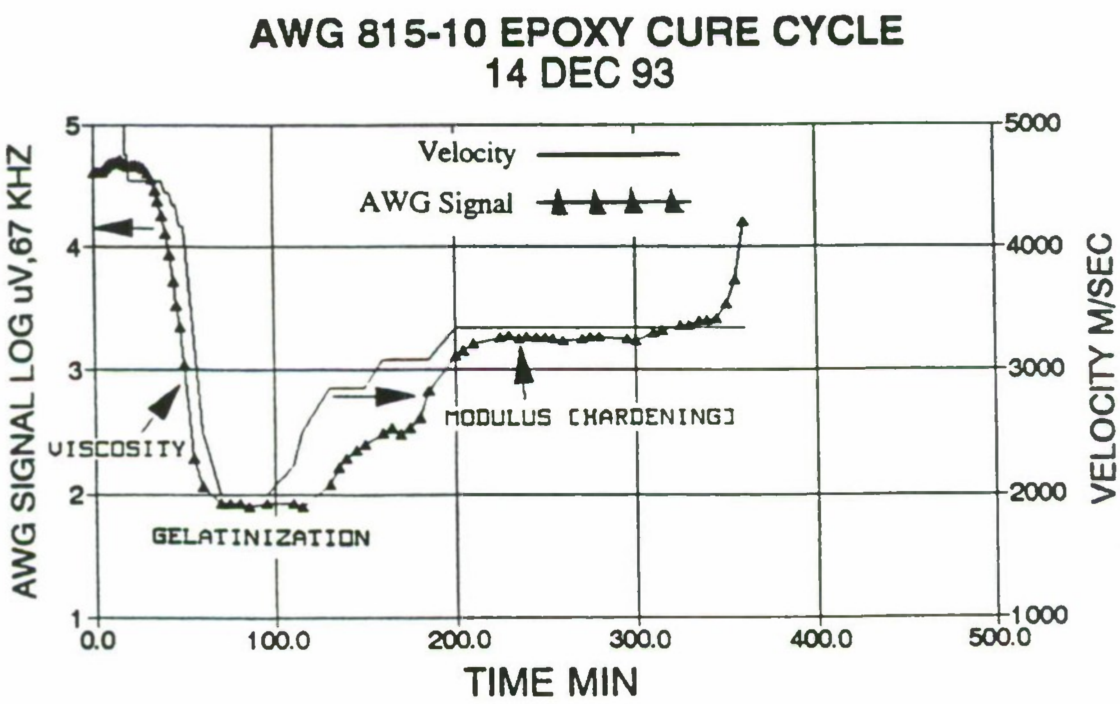


Figure 5-8. Acoustic waveguide cure curves for parts with two right-angled bends molded using Shell 815 resin with accelerator and heated via infra-red.

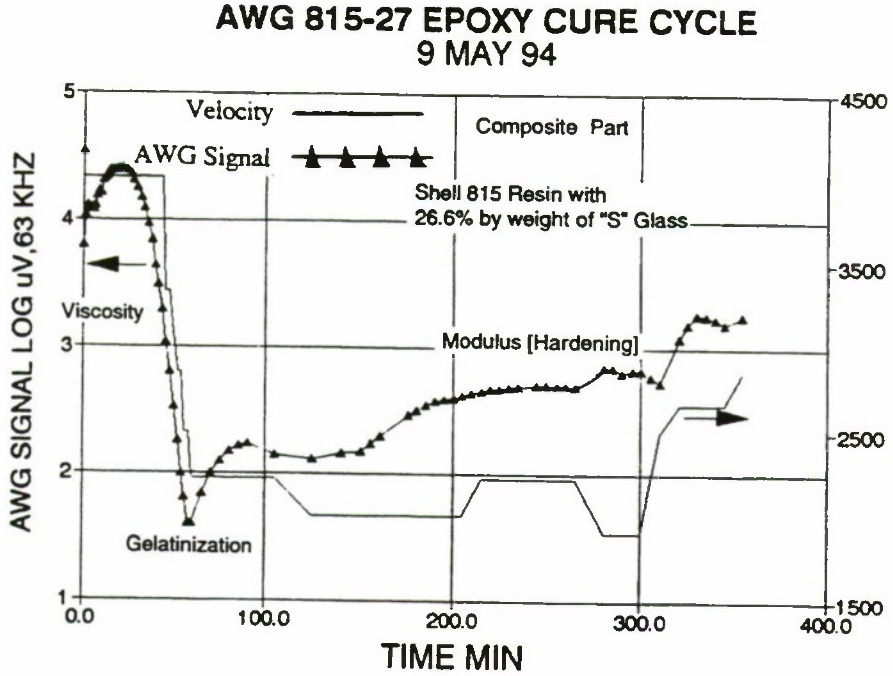
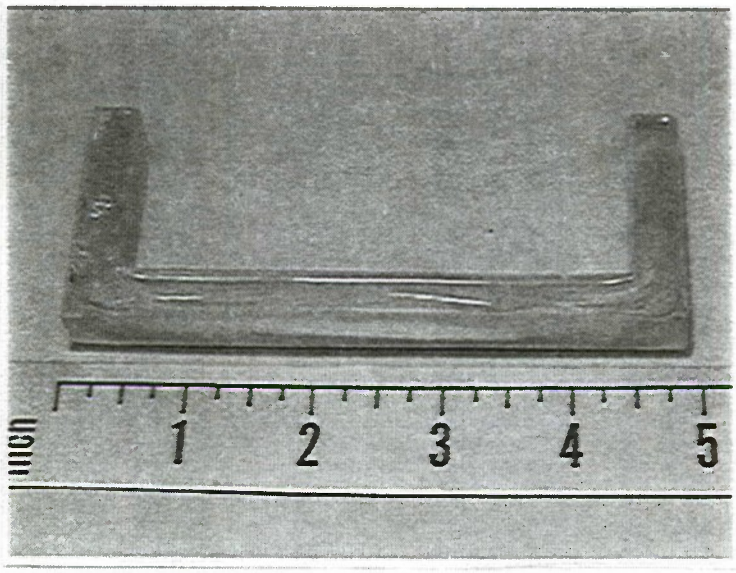


Figure 5-9. Acoustic waveguide cure curve for composite part



Part of Shell 815 resin loaded with
33% by weight of "S" glass fibers.

Figure 5-10. Part of Shell 815 resin re-inforced with "S" glass fibers and cured at room temperature

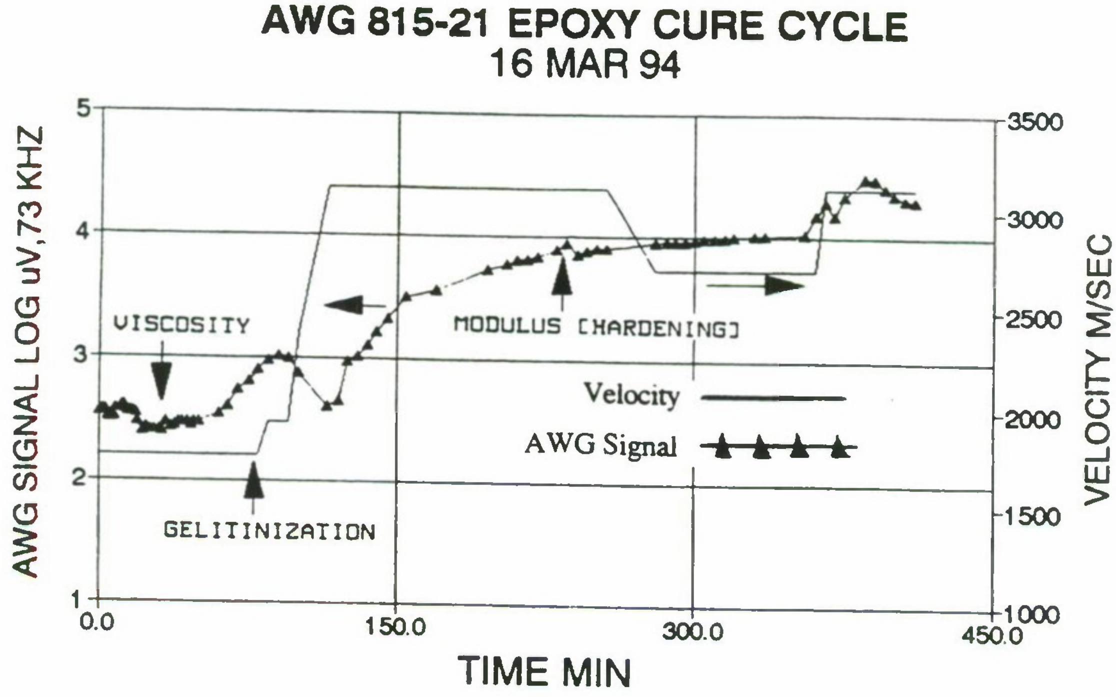
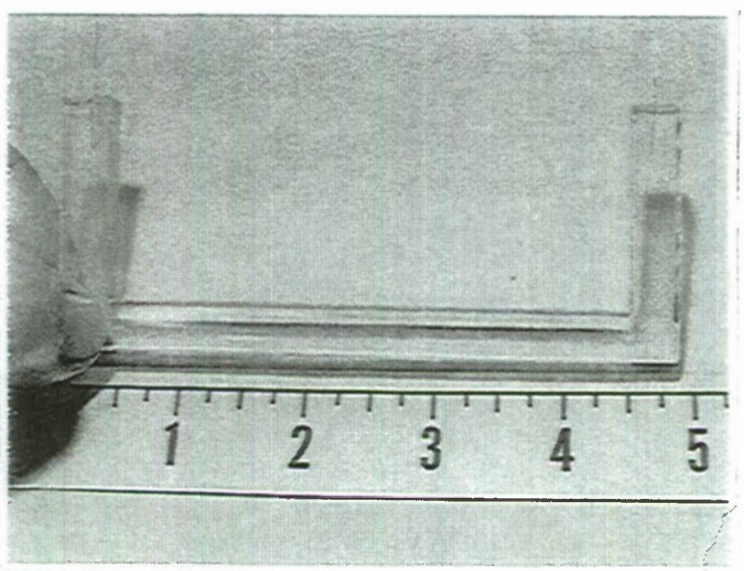


Figure 5-11. Acoustic waveguide cure curves using parts as waveguide (AWG stubs each end of mold)



AWG [10 mil Nichrome] of 5 cm. length penetrate 1 cm. into opposite ends of Shell 815 resin part so that part acts as a gradually forming AWG during cure.

Figure 5-12. AWG inserted into opposite ends of Shell 815 resin part allows part to function as an AWG during cure

signal transmission will take place until a slight hardening of the resin occurs. A picture of a cured resin part with AWG stubs at the ends is shown in Figure 5-12.

5.4.6 Sensing of Voids

Experiments were carried out using Shell 815 resin, room temperature cured over a 50 hour time period, to determine if the presence of voids could be identified by resonances in the acoustic waveguide (AWG) spectrum signature. Two experiments were performed simultaneously using 10 mil diameter Nichrome AWG embedded within 10 cm^3 volume of curing resin contained within 10 cm long, 1.25 cm diameter semi-circular silicone rubber channels, Figure 5-13. One channel contained 0.1 cm^3 volume (~1% of resin voluume) of hollow ceramic spheres (3M Marcolite ceramic spheres 0.6 to 1.4 mm diameter) to represent internal voids which could form during curing.

In Figure 5-14 the acoustic waveguide spectrum signatures (0 to 200 kHz) are shown of room temperature curing Shell 828 resin with and without artificial voids taken 3 hours after pouring the resin, and before gelation which occurred around 25 hours. Both of the two spectrum pictures show the main transmitted acoustic signal at around 64 to 68 kHz, but the signature for the resin with embedded voids clearly indicates resonant frequencies at ~70 kHz, 100 kHz, 110 kHz, 130 kHz and 160 kHz. These resonances are still evident in the signatures recorded 50 hours after pouring the resin, Figure 5-15. Based on a "void" diameter of ~1 mm ($\lambda/2$), where λ is wavelength, an acoustic wave velocity (V) of 330 m/sec for air within the artificial voids, then from $V = f\lambda$, a resonant frequency of 165 kHz can be calculated. This is encouraging and infers that voids occurring during resin curing may be identifiable from the AWG acoustic signature.

Related studies dealing with gas and bubbles within curing resin are outlined in Section 5.6; Acoustic Waveguide Sensing of Gas and Bubbles in Curing Resin.

5.4.7 Modulus-Type of Measurements

"Generally, composite materials* provide a similar strength to metals with a much lighter weight. The role of the fibers (glass or graphite, for example) in a composite is to provide stiffness and strength, and carry in-plane loads, while the matrix (resin, for example) bonds, protects and hold the fibers in place, provides transverse strength and acts as a load transfer medium." Typical properties of composites (*from Norman J. Johnston, NASA) are listed in Table 5-1. In particular, from Table 5-1 it should be noted that the tensile moduli for organic polymers or resins are around 0.5 Msi (3 GPa) and for "S" glass 13 Msi (90 GPa).

Obviously, the tensile modulus is an important property of composite materials and in carrying out modulus-type of measurements, in order to save time and cost, we used acoustic measurements to obtain relative values for the tensile moduli of both resin and composite specimens.

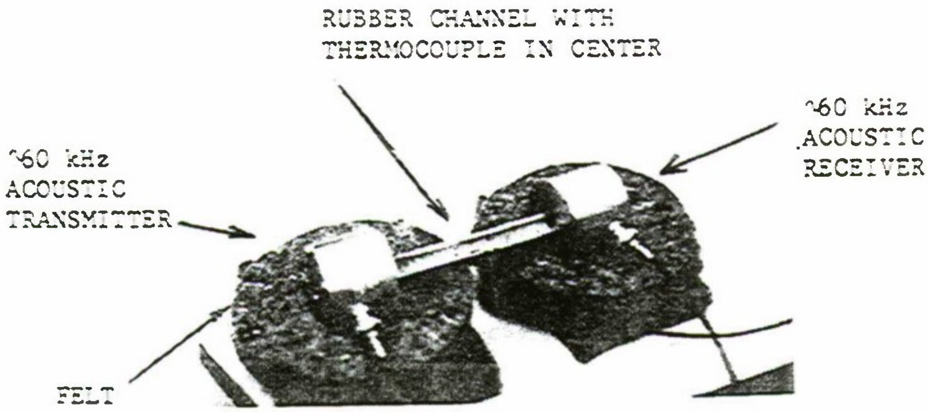
The measurement procedure was to bond acoustic transmitters and receivers to opposite ends of a specimen, transmit longitudinal acoustic waves through the specimen and measure the wave velocity. The theory is that the acoustic wave velocity (C) is related to the density (ρ) and Young's Modulus (Y) of the material. The relationship is:

$$C = \sqrt{\frac{Y}{\rho}} \qquad (1)$$

Table 5-1
*Typical Properties of the Constituents of Continuous Carbon Fiber Reinforced Composite Materials Versus Metals

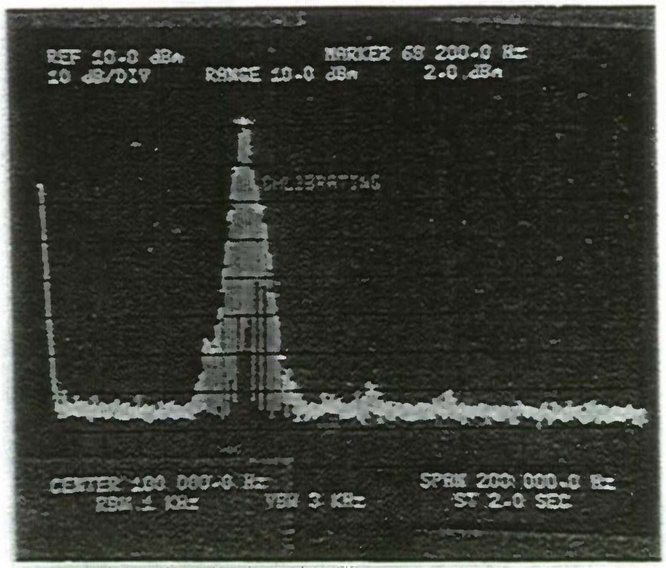
FIBER	REINFORCEMENT TENSILE STRENGTH, Ksi	REINFORCEMENT TENSILE MODULUS, Msi (Gpa)	TENSILE STRAIN, %	DENSITY, g/cc
Carbon	600	34 (230)	1.6	1.8
Carbon (Intermediate Modulus)	800	44 (300)	1.8	1.8
S Glass	665	13 (90)	5.4	2.5
ARAMID	550	19 (130)	---	1.5
Boron	510	60 (400)	0.8	2.5
PE	435	25 (170)	2.7	0.97
	MATRIX			
Organic Polymers	15-20	0.4-0.6 (3)	1.60	1.2-1.4
	METALS			
Steel	70-150	30 (200)	----	7.8
Titanium	125	16 (110)	----	4.5
Aluminum	70	11 (75)	----	2.7

*(These data from Norman J. Johnston, NASA Langley Research Center, Hampton, VA Nov 1992)

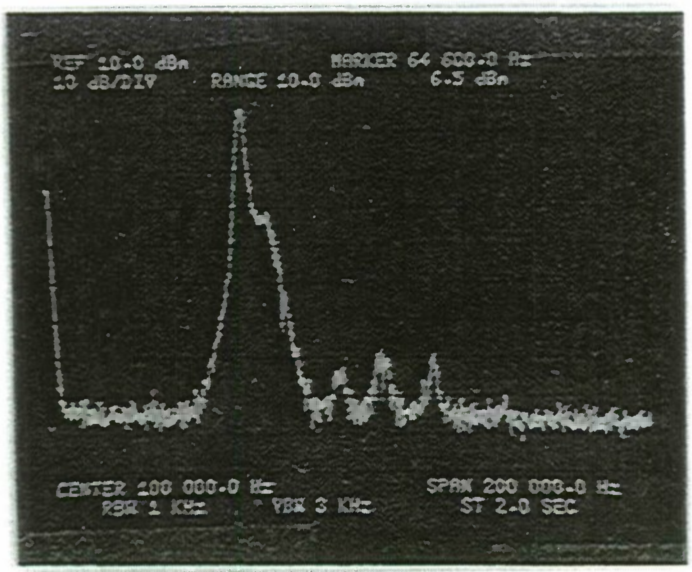


Rubber channel into which resin is poured

Figure 5-13. Experimental apparatus for measuring the acoustic waveguide spectrum signatures of curing resin with and without artificial voids

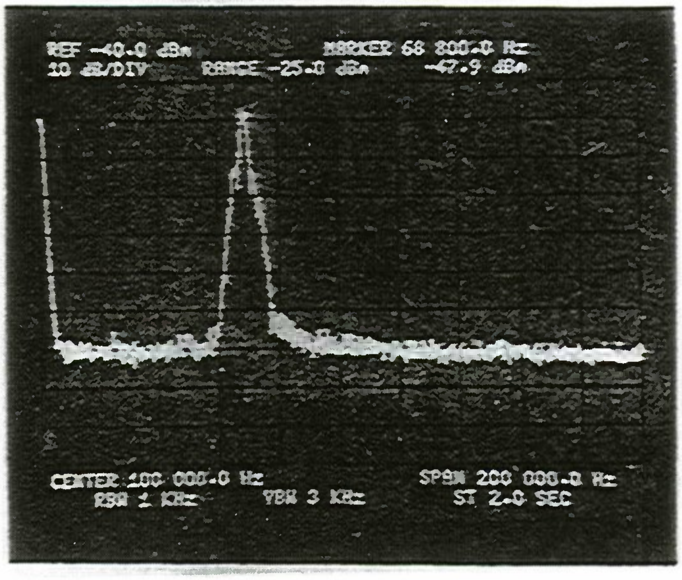


Spectrum signature of curing resin (180 minutes)
without artificial voids

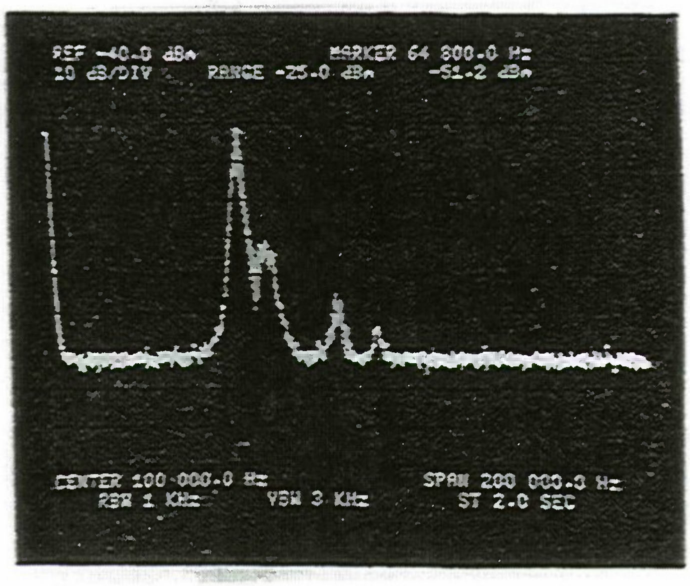


Spectrum signature of curing resin (180 minutes)
with artificial voids ~1% by volume

Figure 5-14. Acoustic waveguide spectrum signatures of room temperature curing resins Shell 828 with and without artificial voids taken 3 hours after pouring resin



Spectrum signature of cured resin (3200 minutes)
without artificial voids



Spectrum signature of cured resin (2750 minutes)
with artificial voids, ~1% by volume

Figure 5-15. Acoustic waveguide spectrum signatures of room temperature cured Shell 828 resins with and without artificial voids taken 50 hours after pouring resin

Acoustic and density measurements were made and the resin parts with two right-angled bends had densities of ~1.14 gm/cm^2 and wave velocities of ~2200 m/sec. While for the part reinforced with 26.6% by weight of "S" glass fibers the respective values were ~1.24 gm/cm^3 and 4100 m/sec; and for the part with 33% of "S" glass fiber, ~1.28 gm/cm^2 and ~5500 msec. From these data, using Equation (1), the calculated moduli values are as follows:

Table 5-2
Moduli Calculated from Acoustic Wave Velocities

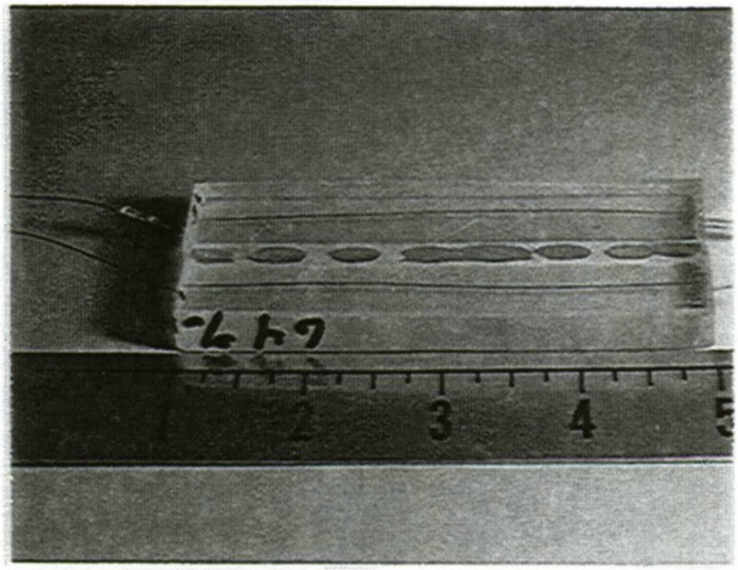
Material	Young's Modulus (GPa)
Shell 815 Resin	5.5
Shell 815 Resin/26.6% "S" Glass	21.0
Shell 815 Resin/33% "S" Glass	32.0

5.4.8 Bondline Integrity

A technical challenge in materials science is the bonding together of parts, especially where complex shapes and cylindrical structures are involved. One problem is that parts which have the appearance of being well bonded, or were processed to be well bonded, may have very little mechanical strength. These types of false bonds are called "kissing bonds". In applying the embedded AWG to the kissing bond problem it was thought that acoustic wave transmission between two AWG, one each side of a bondline could be used to assess the degree of bonding. Also, if the AWG were positioned parallel to, and close to (within one or two cm, or a wavelength) the bondline, then the signal transmission level along a single AWG may also respond to the bondline strength or degree of bonding.

In order to test this hypothesis, resin parts were molded with bondlines of varying bonded areas equispaced between two AWG embedded parallel to the bondline. This was achieved by first molding resin parts 1 x 2 x 10 cm with a 10 mil diameter Nichrome AWG passing through the center in the longitudinal direction, and then partially joining these parts with different areas coated with resin to form varying bondline adhesion areas, Figure 5-16. This configuration allowed acoustic interrogation of the bondline area, not only across the bondline from AWG to AWG, but also by transmission along each AWG located parallel to the bondline. Many interrogation paths are available, for example, using the AWG terminations marked 1,2,3, and 4 in Figure 5-16, acoustic transmission through the bondline can be from 1 to 4, 4 to 1, 3 to 2, 2 to 3, 2 to 4, 4 to 2 and from 1 to 3 and 3 to 1. Interrogation parallel to the bondline can be from 1 to 2, 2 to 1 and 3 to 4, 4 to 3. These types of measurements were made on twelve pairs of specimens bonded together with different degrees of bonding. The bonded areas ranged from zero bond (surfaces pressed together with an acoustic coupling agent, called Aquagel), to several different bonding areas with between 15% to 95% of the total area between parts bonded. The bonding was not precise as it was carried out by applying small drops of resin equispaced along the resin surfaces which were then pressed together and allowed to cure at room temperature. The areas covered by the resin spread as the parts were pressed together and after cure, as the resin is transparent the bonded areas were visually estimated. Next, acoustic interrogation measurements of the various bondlines of different bonding areas were carried out.

The maximum, average and minimum values of the acoustic data for transmission across the bondlines from AWG to AWG, are plotted against, the percentage of bonded area in Figure 5-17. Following the acoustic measurement, mechanical shear strength tests using the apparatus



View showing actual bonded area

1. 2.

3. 4.

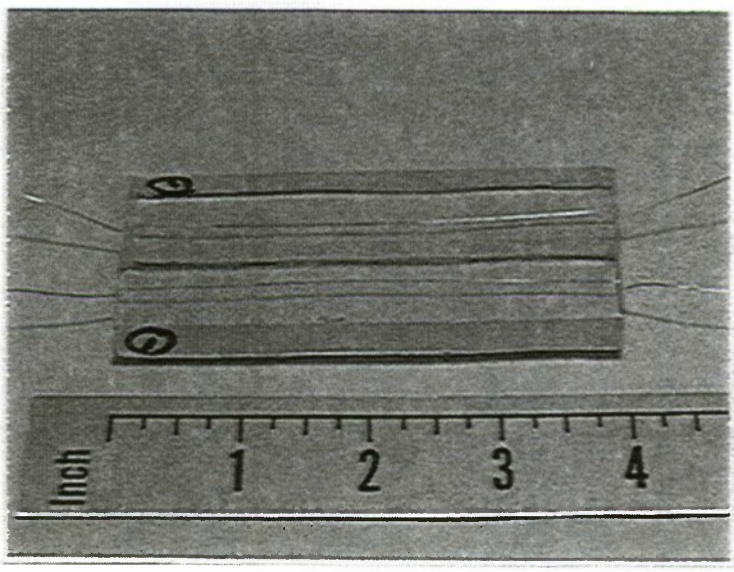


Figure 5-16. Two resin parts, each with an embedded AWG, bonded together

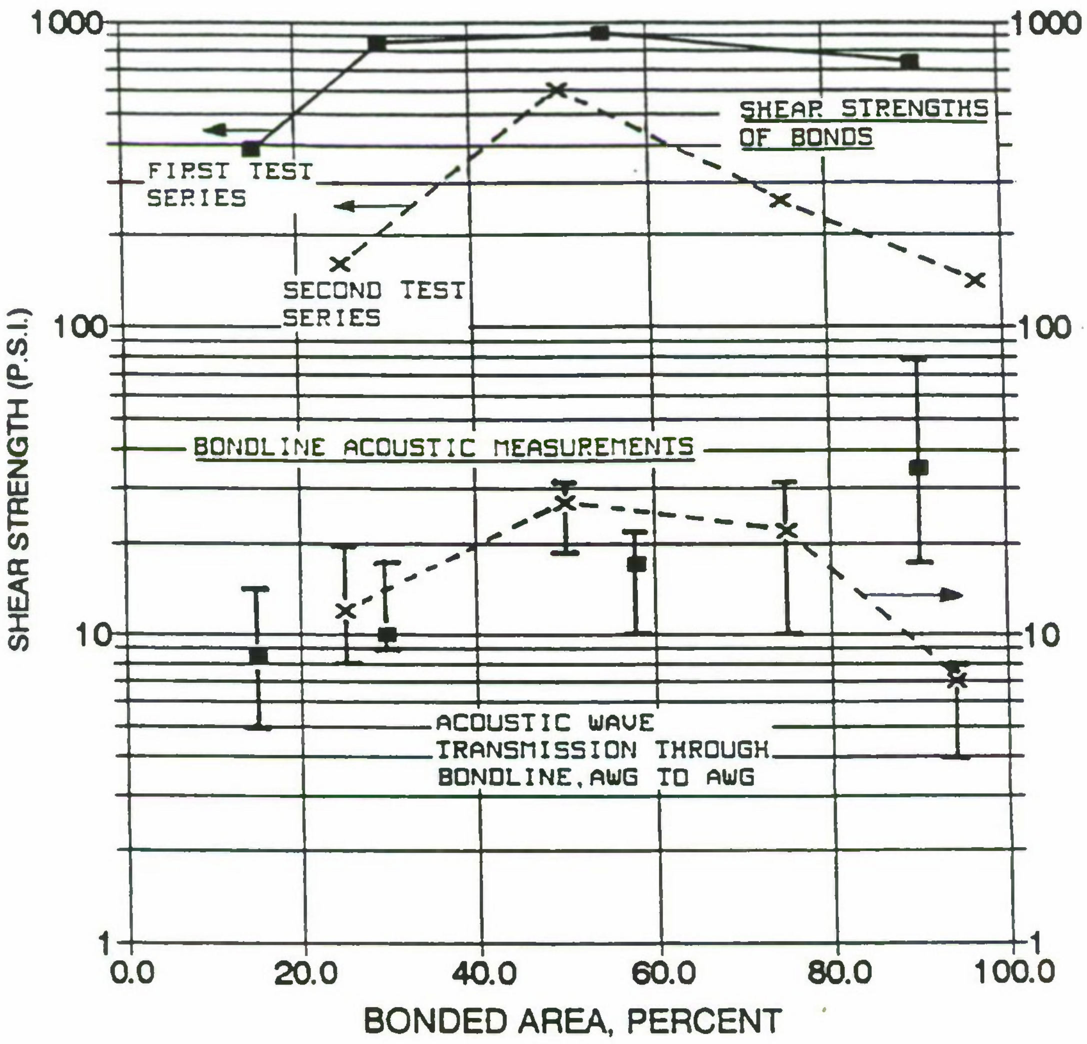
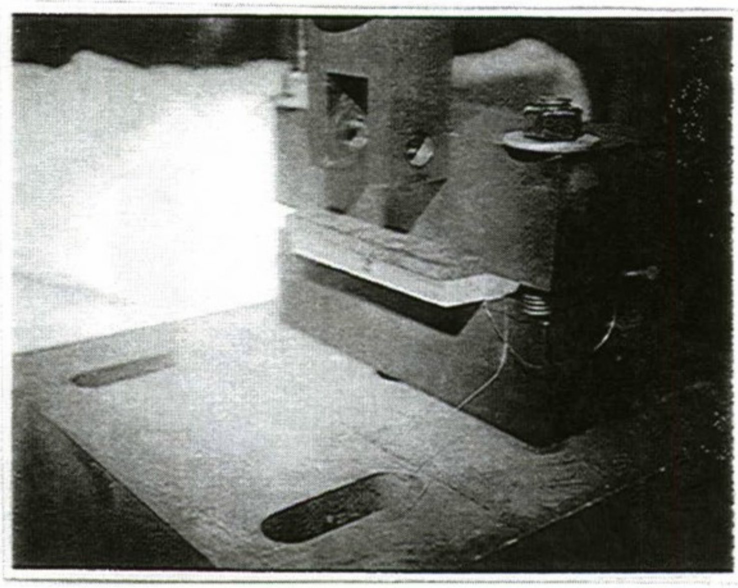
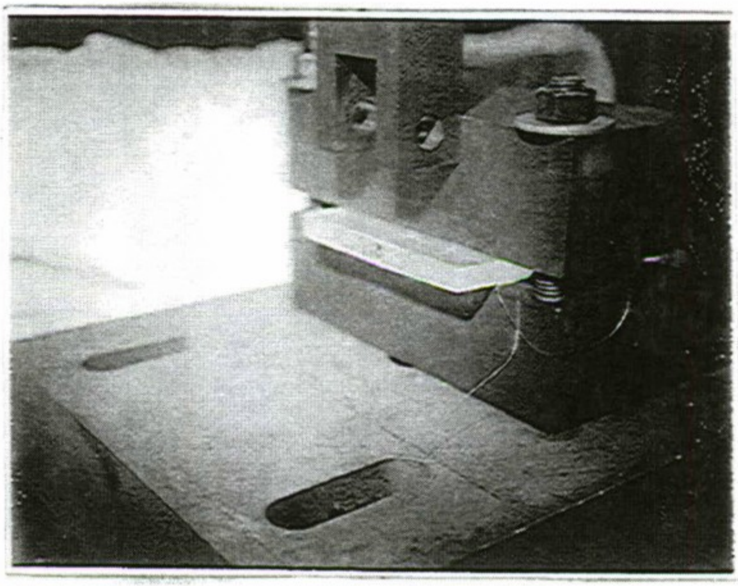


Figure 5-17. Comparison of bondline shear strength and measurements of acoustic wave transmission through bondline, AWG to AWG, versus percent bonded area for pairs of resin parts bonded together.



Steel plate is placed on specimen surface prior to shear strength test.



Resin specimen clamped in position for testing.

Figure 5-18. Bonded resin specimen clamped in position for shear strength test

illustrated in Figure 5-18 were performed on all the bonded specimens and these data are also plotted in Figure 5-17. The most striking result which is apparent in Figure 5-17 is that resin parts with ~50% to ~70% of the area between them bonded have maximum shear strength, while parts with greater or lower percent bonding areas have less shear strength. Furthermore, most of the acoustic bondline measurement data correlates with the shear strength data.

Although these were encouraging results for demonstrating that AWG acoustic interrogation of bondlines can yield credible information in regard to the actual shear strengths of "kissing-type" of bonds, much more experimental confirmatory measurements are required. In addition, the fact that resin specimens with bondlines ~50% to ~70% bonded yielded maximum shear strengths, raises fundamental questions in regard to the bonding of composite materials. Preliminary analysis of the fractured specimens suggests tensile failure of the bonds and not shear failures. It is thought that this was due to the brittle or glassy nature of the resin used for bonding the parts together. Consequently, the tests were repeated using a less glassy bond in order to obtain true shear failures.

In order to obtain a less glassy bond two sets of resin specimens were bonded to varying degrees using a "soft" bonding material, epoxy adhesive Fusor 305 mixed in the ratio one part 305-1,two parts 305-2. This was to ensure that during mechanical strength tests the specimens would fail in shear. The AWG interrogation data for these test series, #7 and #8, are given in Tables 5-3 and 5-4 respectively, while the mechanical shear strength data for both series are listed in Table 5-5. The acoustical and mechanical data are plotted versus percent bonded area in Figures 5-19 and 5-20 respectively. It can be seen from these Figures that acoustical and mechanical data only correlated up to around 60 to 70% of bonded area and not thereafter. This represents a fair degree of correlation and further investigation is required to understand the reason for the lack of correlation in the 70% to 100% bonded area range.

In order to learn more about the acoustic characteristics of resin bondlines, some resin specimens were sawn through to varying degrees to simulate the conditions of material 100% bonded, to material with zero bonding. Measurements of acoustic wave velocity and the transmitted signal level were made at each stage as the depth of the sawcut progressed, for specimens both with and without embedded waveguides. In Figure 5-21 a specimen with an embedded acoustic waveguide and having a 1 mm wide, 3 mm deep sawcut can be seen. As the specimen cross section is 10 mm by 10 mm, and the sawcut depth is 3 mm, then the area of material remaining intact is 4 mm x 4 mm, or 16% of the original area.

For the transmitted signal levels, values are plotted against the percent area intact (or simulated bond area) in Figure 5-22. It can be seen that for the specimen without an embedded waveguide the level of acoustic signal transmitted is directly proportional to the material cross sectional area. While for the specimen with an embedded waveguide, the signal level transmitted through the waveguide is proportional to the square root of the material cross sectional area.

As can be seen from Figure 5-22, when the specimen without an embedded waveguide was completely sawn through, and the two ends were touching (in a dry condition), the transmitted signal level was only 13.6 mV, compared with 1050 mV signal when the material was 100% intact and no cuts have been made. When the dry ends were coupled with an acoustic couplant (Aquagel), the transmitted signal level increased to 96 mV, but still far short of the original 1050 mV.

With the specimen having an embedded waveguide, the maximum sawcut depth (3 mm) around the waveguide left 16% of material intact. This condition resulted in a 2.4 mV signal being transmitted through the waveguide compared with 6.2 mV before any slot was cut in the specimen. With water or Aquagel filling this slot there was negligible change in the transmitted signal level. For the specimen without an embedded waveguide, when the two ends were bonded together with 815 resin, the transmitted signal level returned to its original 1050 mV. When the slot around the

Table 5-3

Data for AWG Interrogation of Bondlines for Test Series #7

Three series of measurements in mV for transmission AWG to AWG. For example, in Figure 5-16, from 1 to 4, 4 to 1; and 2 to 3, 3 to 2.

Receiver #.	#1	#2	#3	AVERAGE	% BOND	FINAL AVE
200	11.4	11.6	11.7	11.57	21	
00-4	4	4.4	5.2	4.53	21	8.18
203	10	8.8	8.2	9	21	
200	8.6	7.6	6.6	7.6	21	
183	9.4	9.2	8.7	9.1	54	
194	21.5	20	18.5	20	54	22.78
216	26.5	29.5	28	28	54	
188	28	32.5	41.5	34	54	
201	31.5	23	22.5	25.67	58	
188	27	17	15.5	19.83	58	16.92
200	13.6	10.1	13.1	12.27	58	
203	10.5	9.6	9.6	9.9	58	
183	8.6	6.6	3.6	6.27	84	
194	11.2	9	6.8	9	84	8.66
202	14.2	7.2	8	9.8	84	
198	15.5	6.6	6.6	9.57	84	
201	32.5	34	30	32.17	95	
188	31.5	32	22.5	28.67	95	16.76
200	2.9	2.65	2.2	2.58	95	
203	2.7	4.1	4	3.6	95	

Another measurement series AWG to AWG, but 1 to 3, 3 to 1, and 2 to 4, 4 to 2.

Receiver #.	#1	#2	#3	AVERAGE	% BOND	FINAL AVE
00-4	6.2	6.5	6.4	6.37	21	
203	9.1	9.2	9.3	9.2	21	8.33
200	8.4	9.3	9.3	8.93	21	
198	7.6	9.7	9.1	8.8	21	
194	12.2	12.2	11.8	12.07	54	
216	12.5	12.9	12.4	12.6	54	38.54
183	50	52	49.5	50.5	54	
188	80	85	72	79	54	
203	38	42	40	40	58	
201	34	36	32.5	34.17	58	26.18
200	20.5	20	20	20.17	58	
188	9	9.6	12.5	10.37	58	
198	15	17.5	16.5	16.33	84	
183	11	14	15.5	13.5	84	22.17
202	33	23	25	27	84	
194	37.5	25	33	31.83	84	
203	16.5	16	15	15.83	95	
201	9	8.8	8.7	8.83	95	11.13
200	5.2	9.1	9.6	7.97	95	
188	8.1	12.6	15	11.9	95	

This series of measurements along each AWG from 1 to 2, 2 to 1, and 3 to 4, 4 to 3.

Receiver #.	#1	#2	#3	AVERAGE	% BOND	FINAL AVE
198	18	19	19	18.67	21	
00-4	16.5	19	17.5	17.67	21	13.42
203	7.9	7.3	10.5	8.57	21	
200	7.2	9	10.1	8.77	21	
188	44.5	48	46	46.17	54	
194	55	61	59	58.33	54	36.92
216	25	33	23	27	54	
183	14	16.5	18	16.17	54	
201	42	37.5	39.5	39.67	58	
200	95	66	50	70.67	58	38.92
188	27	19	17	21	58	
203	30	20	23	24.33	58	
183	6.6	7.1	8	7.23	84	
202	15	16	13.7	14.9	84	14.87
194	22.5	17.5	17.5	19.17	84	
198	17	18.5	19	18.17	84	
201	5.2	6.8	8.3	6.77	95	
200	10.8	13.1	18.5	14.13	95	6.48
188	2.3	2.5	2.4	2.4	95	
203	2.9	2.4	2.5	2.6	95	

Table 5-4

Data for AWG Interrogation of Bondlines for Test Series #8

Three series of measurements in mV for transmission AWG to AWG. For example, in Figure 5-16, from 1 to 4, 4 to 1; and 2 to 3, 3 to 2.

Receiver #.	#1	#2	#3	AVERAGE	% BOND	FINAL AVE
202	16	17	16	16.3	25	
201	15	16	15	15.3	25	10
194	4.65	4.8	3.7	4.4	25	
195	4.4	4.5	3.6	4.2	25	
203	2.05	3.3	2.8	2.72	26	
200	1.65	2.6	2.3	2.18	26	4.1
182	3	2.8	3	2.9	26	
188	7.4	8.5	9.8	8.57	26	
189	1.1	1.05	1.3	1.15	49	
190	0.72	0.7	0.78	0.73	49	6.85
164	8.3	12.4	10.9	10.53	49	
19	11	18	16	15	49	
195	10.8	8.7	7.6	9.03	67	
202	12.8	10	8.6	10.47	67	16
201	19.5	20.5	24	21.33	67	
194	24	24.5	21	23.17	67	
13	11.3	10.7	10.8	10.93	83	
00-4	10.6	10.9	10.8	10.77	83	9.63
215	7.8	8	6.4	7.4	83	
18	10.1	11.6	6.5	9.4	83	

Another measurement series AWG to AWG, but 1 to 3, 3 to 1, and 2 to 4, 4 to 2.

Receiver #.	#1	#2	#3	AVERAGE	% BOND	FINAL AVE
194	17.5	20	19.5	19	25	
202	18	20	19	19	25	15.5
195	12.2	11.6	13.2	12.3	25	
201	11.3	11	12.4	11.6	25	
188	4	3.6	3.7	3.77	26	
203	2.5	2.3	2.3	2.37	26	3.75
182	3.2	3.4	3.25	3.28	26	
200	5.5	5.8	5.5	5.6	26	
19	2.2	1.7	1.45	1.78	49	
189	2.05	1.95	1.85	1.95	49	4.18
164	7.4	5.7	5.65	6.25	49	
190	7.8	6	6.2	6.67	49	
194	8.2	8	8.4	8.2	67	
195	12	11.7	11.9	11.87	67	21.23
201	32	34	34	33.33	67	
202	28.5	31	35	31.5	67	
18	11.6	12	12.6	12.1	83	
13	14	15	15	14.67	83	29.99
215	67.5	68	78	71.17	83	
00-4	23	22	21	22	83	

This series of measurements along each AWG from 1 to 2, 2 to 1, and 3 to 4, 4 to 3.

Receiver #.	#1	#2	#3	AVERAGE	% BOND	FINAL AVE
202	13.6	15.5	16.2	15.1	25	
195	13.2	14.7	14.8	14.2	25	11.98
201	12	8.6	8.6	9.7	25	
194	11.8	7.4	7.4	8.9	25	
203	1.6	1.4	1.2	1.4	26	
182	1.3	1.36	1.18	1.3	26	6.5
200	9.4	12.4	13.1	11.6	26	
188	9.4	12.4	13.3	11.7	26	
189	7.6	10.8	11	9.8	49	
164	5	6.8	6.6	6.13	49	4.75
190	1.7	1.3	1.5	1.5	49	
19	1.55	1.6	1.5	1.55	49	
195	18.5	14.5	14.8	15.93	67	
201	19	19.5	20.8	19.77	67	20.93
202	18.5	19	19	18.83	67	
194	30	33	24.5	29.17	67	
13	18.5	12.8	12.6	14.63	83	
215	22.5	15	15	17.5	83	12.8
00-4	10.8	10.1	6.6	9.17	83	
18	12	10.7	7.1	9.93	83	

Table 5-5
Data for Mechanical Strength Tests of Bondlines for Test Series #7

WESTINGHOUSE STC NUCLEAR SERVICES &
MATERIALS TESTING LAB

CUSTOMER: R.T. HARROLD

TESTED BY: E.J. HELM, Jr.

TEST TYPE: SHEAR OF BOND

TEST MACHINE: 50 KIP INSTRON
SERIES 7
CHARGE NO.: 9TD1-ACTAC-08 DATE: 9/30/94

% BOND	FAILURE LOAD [POUNDS]	LENGTH Inches	WIDTH Inches	AREA [SQ. IN.]	SHEAR STRENGTH [PSI]
21	327.2	3.940	0.411	1.619	202.058
54	738.5	3.950	0.445	1.758	420.139
58	572.4	3.962	0.412	1.632	350.661
84	1650.8	3.980	0.455	1.811	911.591
95	1750.4	3.985	0.430	1.714	1021.505

WESTINGHOUSE STC NUCLEAR SERVICES &
MATERIALS TESTING LAB

CUSTOMER: R.T. HARROLD

TESTED BY: E.J. HELM, Jr.

TEST TYPE: SHEAR OF BOND

TEST MACHINE: 50 KIP INSTRON
SERIES 8
CHARGE NO.: 9TD1-ACTAC-08 DATE: 9/30/94

% BOND	FAILURE LOAD [POUNDS]	LENGTH Inches	WIDTH Inches	AREA [SQ. IN.]	SHEAR STRENGTH [PSI]
25	306.7	3.970	0.414	1.644	186.605
26	483.5	3.970	0.425	1.687	286.561
49	798.0	3.960	0.430	1.703	468.640
67	1378.3	3.960	0.420	1.663	828.704
83	1608.8	3.960	0.416	1.647	976.593

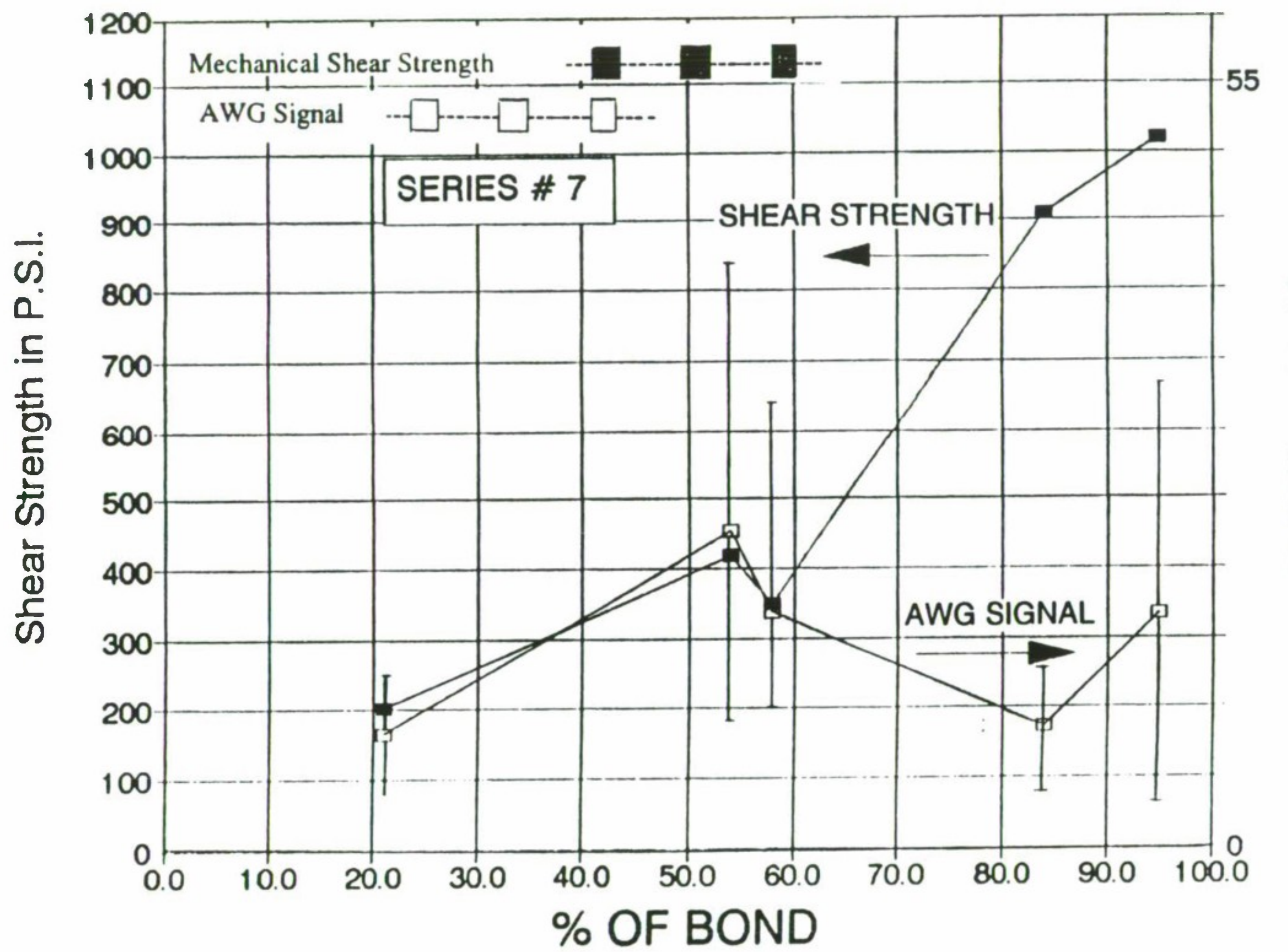


Figure 5-19. Comparison of bondline shear strengths and measurements of acoustic wave transmission through bondlines, AWG to AWG, versus percent bonded area for pairs of resin parts bonded together. SOFT Bond Test Series #7

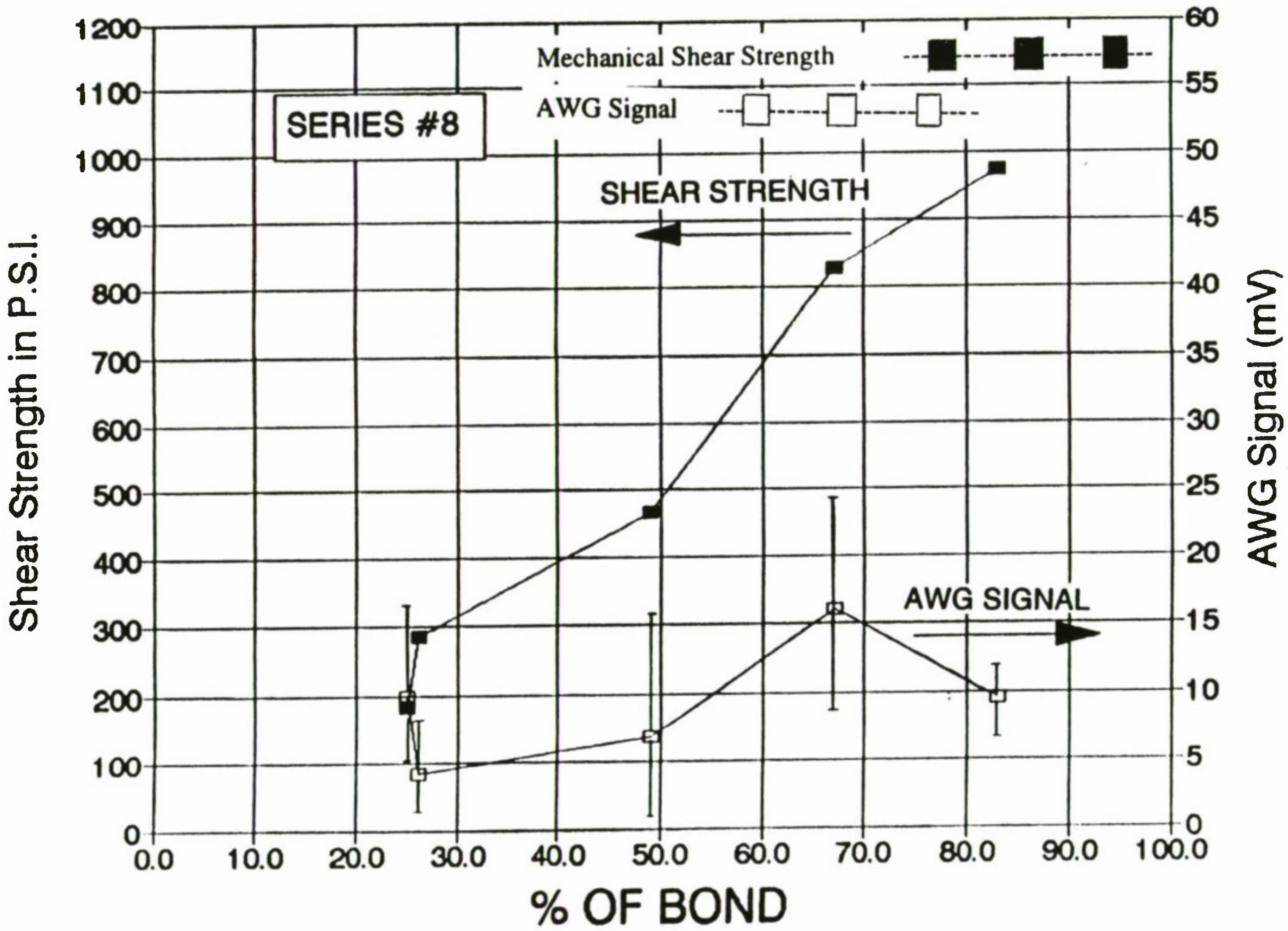
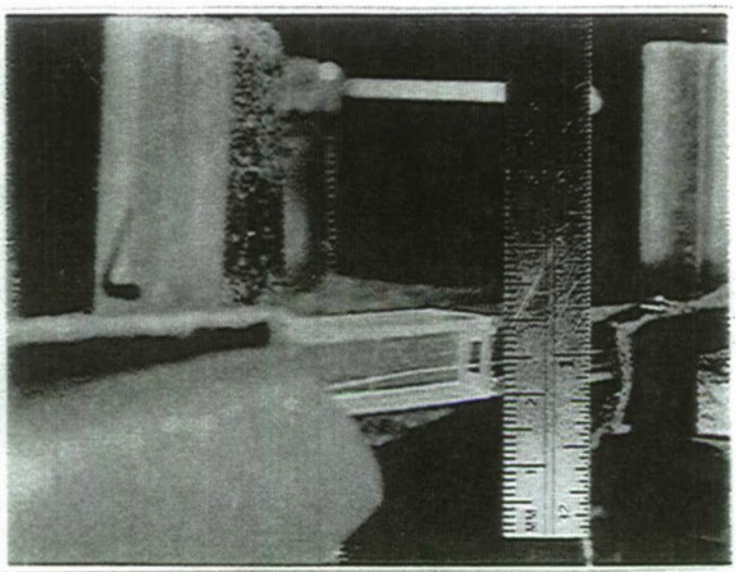
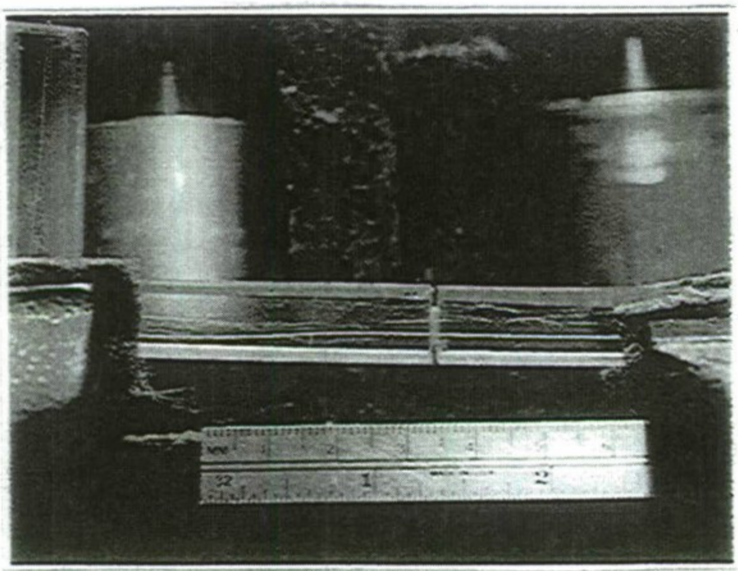


Figure 5-20. Comparison of bondline shear strengths and measurements of acoustic wave transmission through bondlines, AWG to AWG, versus percent bonded area for pairs of resin parts bonded together. SOFTBond Test Series # 8



10 mil diameter Nichrome acoustic waveguide
can be seen passing through intact area



1 mm wide, 3 mm deep sawcut leaves
16% of specimen cross section intact

Figure 5-21. Specimen of 815 resin with embedded waveguide and 1 mm wide, 3 mm deep sawcut to simulate degree of bonding

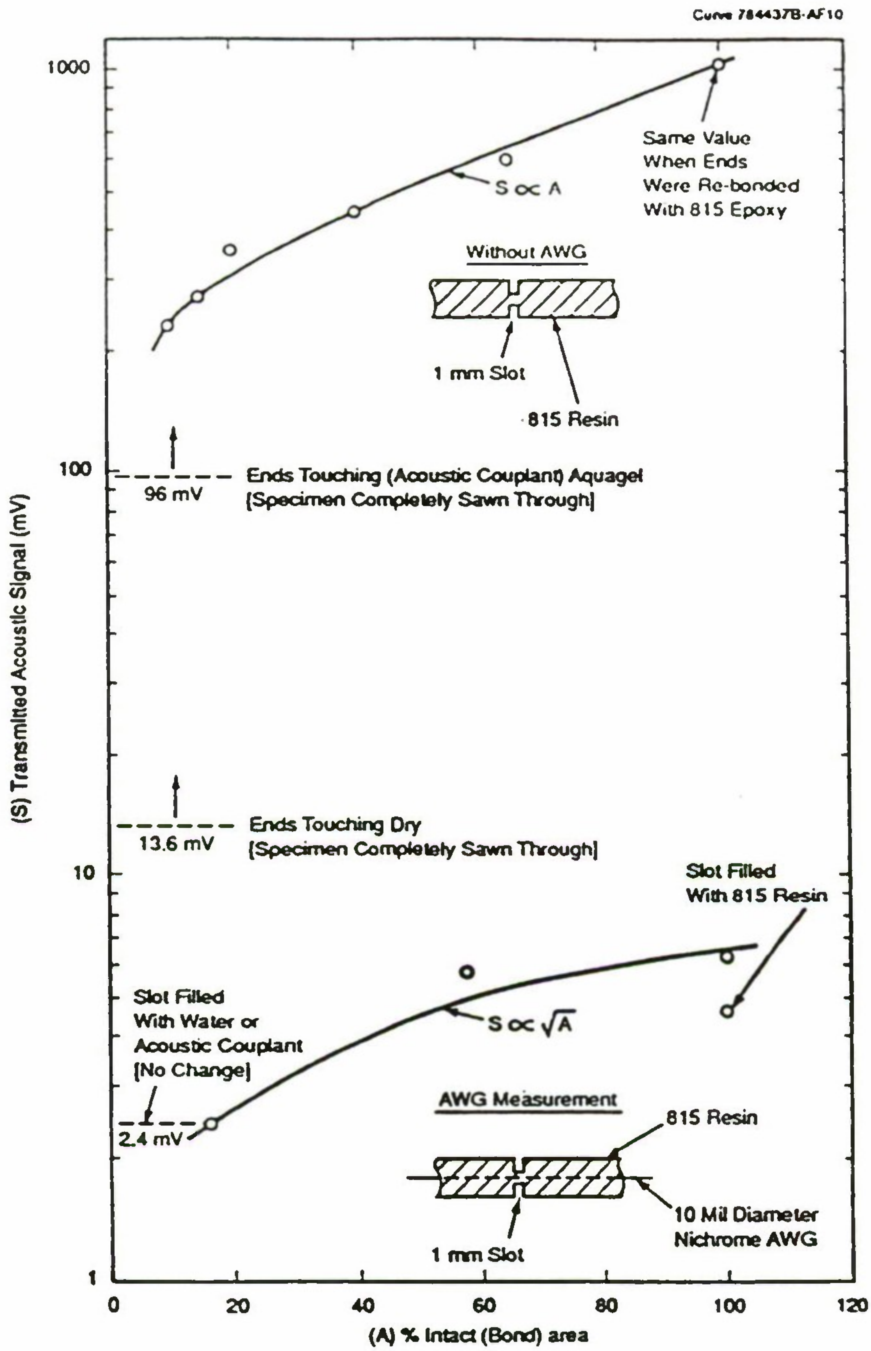


Figure 5-22. Acoustic signal transmission versus "bond area" for resin specimens (with cut slots to simulate reduced bonding) both with and without embedded waveguides

embedded acoustic waveguide was filled with cured 815 resin the transmitted signal level reached ~5 mV, but did not return to the original 6.2 mV level.

It is believed that these data are important because a single AWG embedded within a composite material would not only have the potential to monitor cure and lifetime NDE, but also be able to measure bondline integrity. For example, for inspection of a composite cylinder with a reinforcing ring bonded inside, an AWG could be embedded circumferentially either within the cylinder or the ring.

Measurements of bondline integrity were repeated using a resin specimen with degrees of bonding simulated (by sawcuts) and with an AWG passing through the bond area. This specimen was examined for both peak pulse and spectral (40 to 80 kHz) transmission characteristics in both directions along the embedded AWG, from termination "A" to termination "B", and from "B" to "A". This is different to the previous measurements in which no spectral measurements were made and peak pulse transmission was in one direction only.

The results illustrated in Figure 5-23 show decreasing transmitted signal levels with increasing depth of the sawcut in the resin surrounding the AWG. The reason for using a sawcut-type of slot in the resin was to create a reflection site for the AWG transmitted signal and simulate the acoustic characteristics of a kissing bond for example, in which apparent good physical contact at the bond site has poor acoustic transmission characteristics due to acoustic wave reflections. In Figure 5-23 it can be seen that for zero bond, when the resin is completely cut through, and the only acoustic transmission at the bond site is via the AWG, then the acoustic signal level (compared with 100% bond) has reduced by 200 to 1. It should also be noted that the peak pulse measurements in Figure 5-23, which indicate different signal levels at opposite terminations are linear measurements, while the spectral measurements are logarithmic. Consequently, the spectral measurements, although appearing to have similar values at each termination, are different, for example, 6dB difference is actually 2 to 1.

A noteworthy feature that can be seen in the spectral records is a double hump in the second spectral peak for the 64% and 16% bonds, and this is not evident in the case of 100% bond. It is possible that this double hump is related to acoustic reflection at the bondline.

These analyses of the AWG response to simulated degrees of resin bonding is considered proof-of-principle for embedded AWG interrogation of bondlines and the key measurement values are summarized in Table 5-6.

Table 5-6
AWG Pulse and Spectral Transmission Characteristics for Simulated Resin Bonds

	Degree of	Received Peak Pulse, mV.			SPECTRUM SIGNATURES 40-80 kHz.			
					Term. "A	Term. "B"		Ratio B/A
Specimen	Simulated Bond	Term "A"	Term "B"	Ave. mV.	AVE. dB	AVE. dBm.	AVE. dBm.	AVE. dB.
815-7	100%	7.5	10.8	9.15	-86.80	-85.53	-86.15	1.27
815-7	64%	6.1	6.8	6.45	-88.13	-86.38	-87.26	1.75
815-7	16%	1.6	2.1	1.85	-92.27	-90.24	-91.26	2.03
815-24/A	0%	0.06	0.07	0.065	-96.5	-94.8	-95.65	1.7

Note: "A" and "B" refer to the terminations of the AWG. AWG signals were transmitted in both directions along the AWG and received at terminations "A" and "B" in sequence.

These proof of principle tests were made under ideal conditions, with no changes in the AWG angle of entry into, and exit from, the resin. Consequently, signal transmission and reception was constant and did not change unless the bondline acoustic characteristics changed. In practice any bends in the AWG at entry or exit will cause changes in the terminal signal transfer

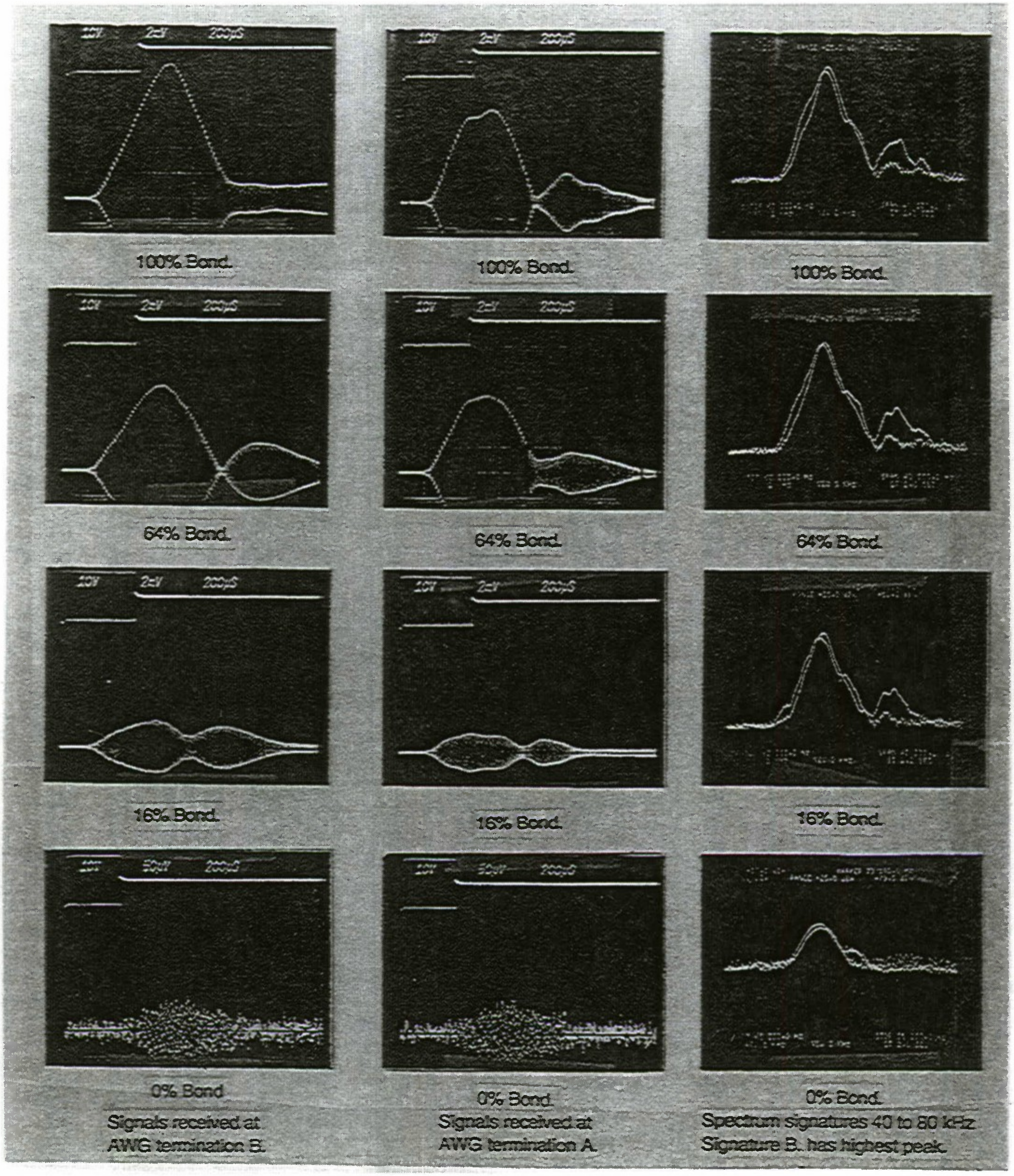


Figure 5-23. Signal levels transmitted in opposite directions through acoustic waveguide embedded in resin specimen and passing through bondlines with degrees of bonding simulated by different depth of sawcuts

characteristics and bondline interrogation would be affected. The challenge ahead in practice is to refine the technique so that changes in the terminal transfer function do not affect the measurements.

5.4.9 Optical Strain Patterns

In order to help in the study of residual strain within molded resin parts a Polariscope was used in which polarized light is beamed through a clear resin specimen to reveal residual strain patterns which are scanned by a TV camera and displayed on a TV screen. An example (picture of TV screen) of residual strain patterns in a right angle bend in resin specimens, both with and without an embedded AWG, is shown in Figure 5-24. In this example it does not appear as though the embedded AWG has caused any internal strain problems. However, much more work is required before conclusions can be drawn. More information on optical strain measurements is given in Section 5.8.

5.4.10 Non-Destructive Evaluation Recommendations

Although the major thrust of this program is directed toward the application of embedded AWG for monitoring liquid composite molding, studies of AWG non-destructive evaluation are included. In particular, these studies include; 5.4.7, Modulus-Type of Measurements; 5.4.8, Bondline Integrity and 5.8, Acoustic Waveguide Sensing of Strain Inside Resin.

Following AWG process monitoring or cure monitoring of a composite part, it is recommended that the embedded AWG first be used for measuring the wave velocity which can give a relative value for the modulus of a part. This would be a quality control application, Section 5.4.7. If bondlines are involved, then acoustic wave transmission from AWG to AWG, across a bondline or along AWG parallel to the bondline can yield information on the bondline integrity, Section 5.4.8. In addition, the embedded AWG can be applied for estimating void content, Section 5.4.6. During field use of a composite part with embedded AWG, the AWG may be applied for internal strain sensing, Section 5.8. Also, in the field, regular AWG modulus measurements could help in damage sensing and remaining life estimates.

With multiple embedded AWG or grid networks, AWG measurements can help in damage location by sonic triangulation, for example. Another field use of embedded AWG is as a listening device to sense and record external damaging impact, and from acoustical spectrum signatures, identify the type of damage. For example, with fiber reinforced composites, different acoustical spectrum signatures occur due to fiber breakage, matrix cracking and delamination.

5.4.11 Acoustic Waveguide Repair

The repair of an AWG, broken where exiting from a resin part, Figure 5-25 was successfully carried out. It was found that when a broken AWG was bonded (with epoxy resin) back onto the resin part, that acoustic waves would travel through the embedded AWG at a velocity close to that existing before the AWG was broken. If the acoustic waves had traveled through the bulk resin and not through the embedded AWG, the wave velocity would have been considerably faster. The data associated with the repaired AWG are listed in Table 5-7. Key measurements are, for part #23, wave velocities of 1370 m/sec in the embedded AWG both before and after repair, and 1600 m/sec in the bulk resin. Another key feature is the level of received signal, i.e., 155 mV to 185 mV for wave travel through the resin, compared with 9 to 11.6 mV for wave travel through the embedded AWG.

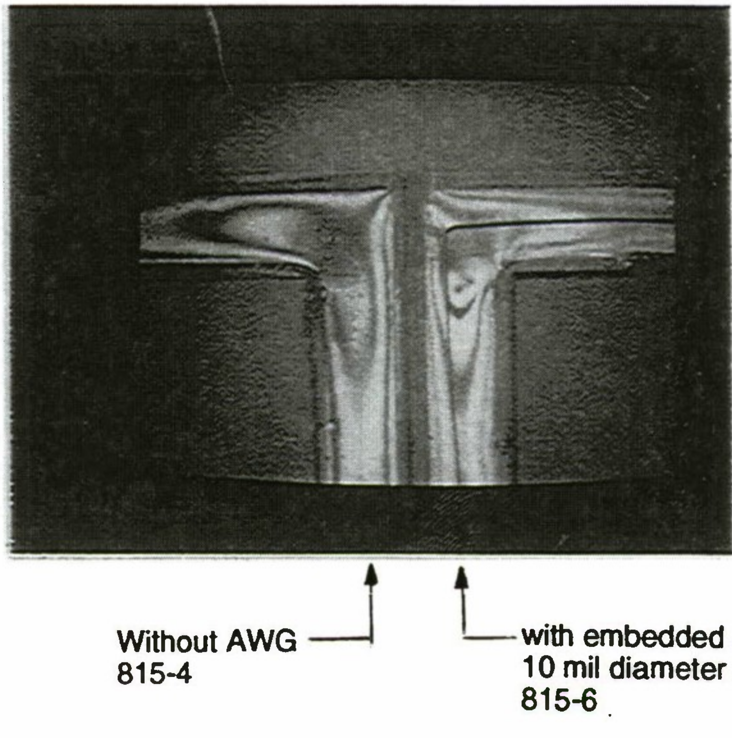
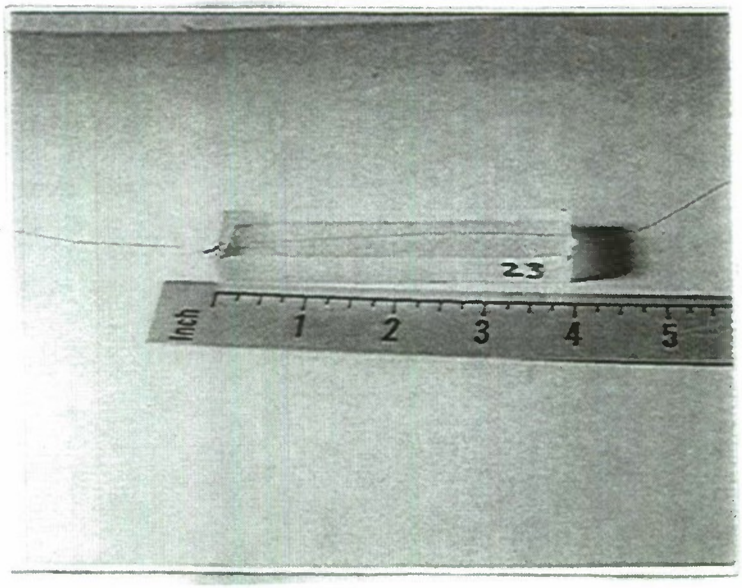
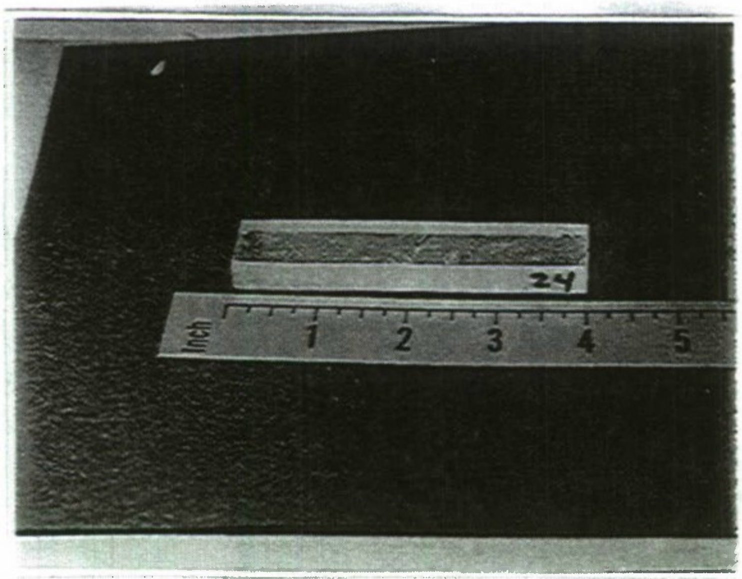


Figure 5-24. Residual strain patterns within molded right-angle specimens of clear Shell 815 resin both with and without embedded AWG

AWG broken off this end and bonded back on with resin



Resin part with embedded 10 mil Nichrome AWG. AWG was broken off one end and bonded back on with resin



Resin part without an embedded AWG

Figure 5-25. Pictures of resin parts, with and without an embedded AWG and used for determining effectiveness of repairing AWG broken off at exit site from resin

TABLE 5-7
Repair of Embedded AWG and Acoustic Wave Velocities and Received Signal Levels:

	Acoustic Wave Velocities			Received Signal Levels		
Part Identification: 815 Resin 10 x 2 x 1 cm	Within AWG	Within AWG After Repair	Within Resin	AWG	AWG After Repair	Resin
#23 with embedded AWG	1370 m/sec	1370 m/sec	1600 m/sec	11.6 mV, 70 kHz 10.0 mV, 66 kHz	8.6 mV, 52 kHz 9.0 mV, 52 kHz	
#23 (Using externally bonded 10 mil AWG each end of resin part. Not aligned with embedded AWG).			1300 m/sec	295 mV, 52 khz 465 mV, 52 khz		
#24 No AWG			1670 m/sec			
#24 Using externally bonded 10 mil AWG each end of resin.			1760 m/sec			185 mV, 55 kHz 155 mV, 55 kHz

5.5 PHASE II - Accomplishments and Results:

The significant accomplishments and results of Phase II - Definition of Military and Industrial Areas, are outlined and described in the following:

5.5.1 Design of Generic Part

The concept of a generic part was to make a liquid molded composite part with embedded AWG which would represent a practical part suitable for both military and civilian automotive vehicles. Following extensive discussions with the TACOM C.O.T.R., Major Richard Brynsvold, a half inch thick part with a curved portion creating a 90° bend was designed, Figure 5-26. This part is representative of a section of the composite armored vehicle (CAV) crew capsule and clear resin was used so as to allow optical strain viewing of resin parts. The reinforcement selected for the composite part was 30 to 40% by weight of either "E" or "S" glass fibers. Acoustic waveguides in some specimens were planned to be at different positions in the composite part as shown in Figure 5-26. The plan was, that as curing of a part generally proceeds with time from the outside surface to the center of a part, then the AWG placement would provide a measure of progressive curing. In addition, the AWG placement would allow for internal measurement of compressive strain, tensile strain and also, a neutral zone measurement. As in Phase I, Section 5.4.1, small resin part, the same weight ratios of resin (65%), hardener (28.5%) and accelerator (6.5%) were used. Typical actual weights were 150 gms of Shell 815 resin, 66 gms of T-403 hardener, and 15 gms of 399 accelerator.

5.5.2 Mold for Generic Part

The mold for the generic part was constructed of Teflon with an open top so that resin could be readily poured into it, Figure 5-27. Later, a polycarbonate front was used on this mold so that resin flow through the mold and "S" glass fibers could be observed. It can be seen in Figure 5-27 that four inserts are placed within the mold to hold and position the AWG. The inserts are made from Shell 815 resin which is the same resin used to make the generic part. When the generic part was made with reinforcing glass fibers the inserts for holding the AWG were not required as the glass fibers supported and positioned the AWG during pouring of the resin.

5.5.3 Cure Curve for Generic Part

Representative cure curves for generic parts (Table 5-8 lists the generic parts) are shown in Figure 5-28. These curves were from a generic part of the same dimensions of that shown in Figure 5-26 but flat, rather than curved. Different shaped generic parts were molded in order to learn about shape differences and acoustic characteristics, Figure 5-29. The three simultaneous cure curves, Figure 5-28, RX1 for the top AWG, RX2 for the center AWG and RX3 for the bottom AWG, all correlate well. The resin viscosity, gelatinization and hardening (modulus) stages are all measured. At final "cure", 380 minutes, or when the last readings were made, the center AWG has the lowest signal level, which infers a lower modulus value in the center of the part as might be expected, because the outer areas of a part should harden first with progressive hardening toward the center of a part with time. AWG cure curves for another flat generic part are shown in Figure 5-30. This part had two 10 mil embedded Nichrome AWG and AWG stubs at the ends, see 5.4.5. It should be noted that the cure curves for the complete AWG give excellent viscosity data at the start of curing while the cure curve for the AWG stubs (acoustic transmission

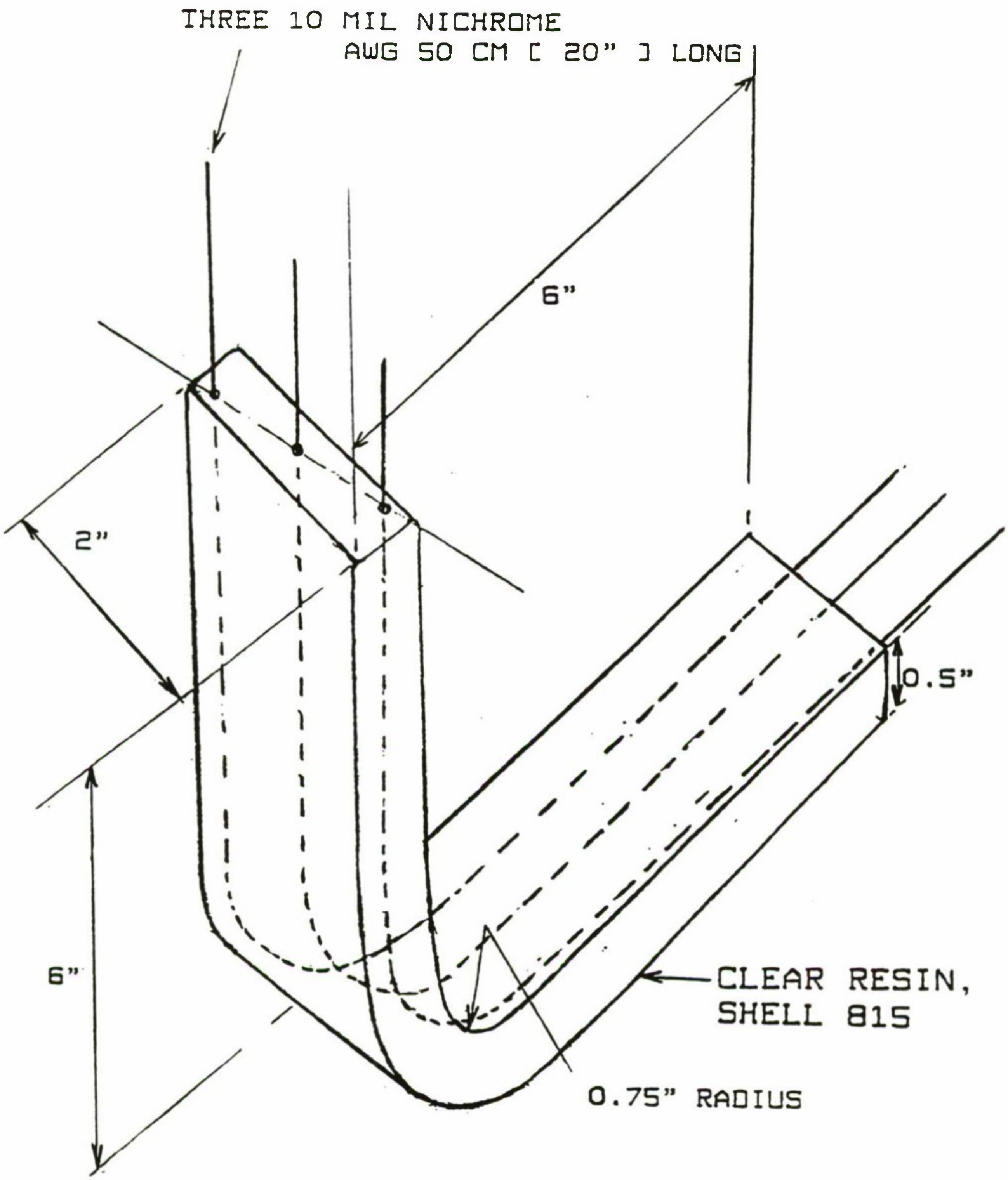
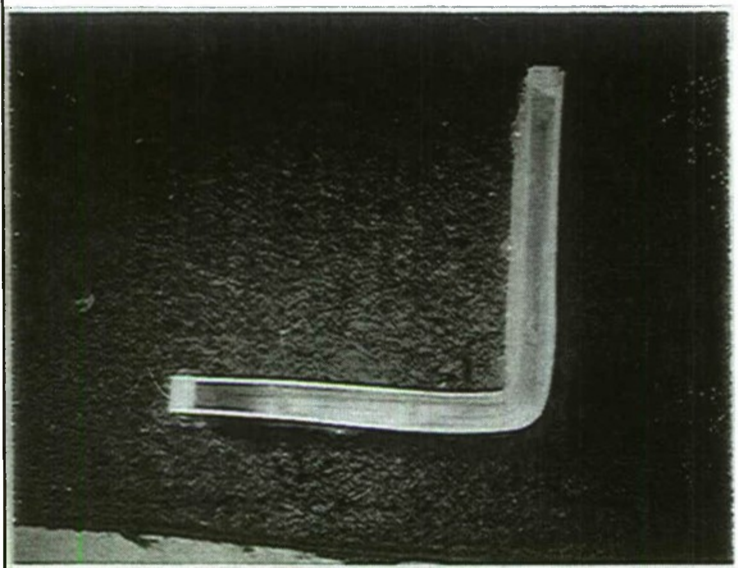
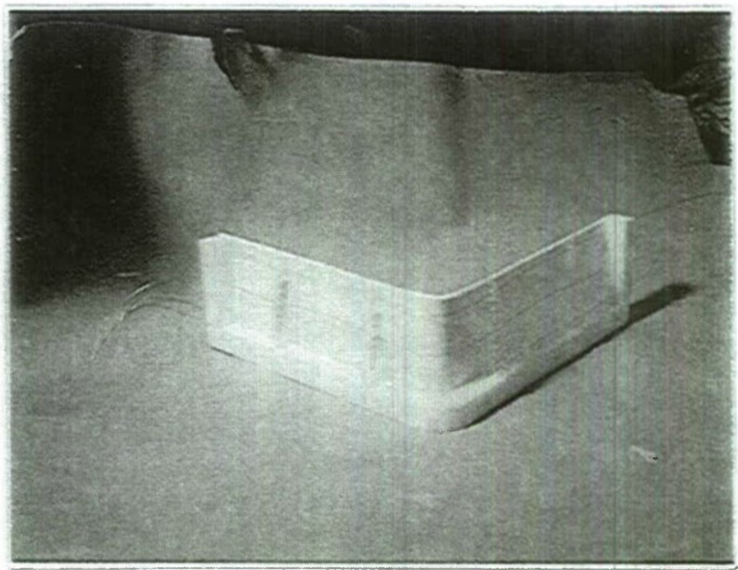


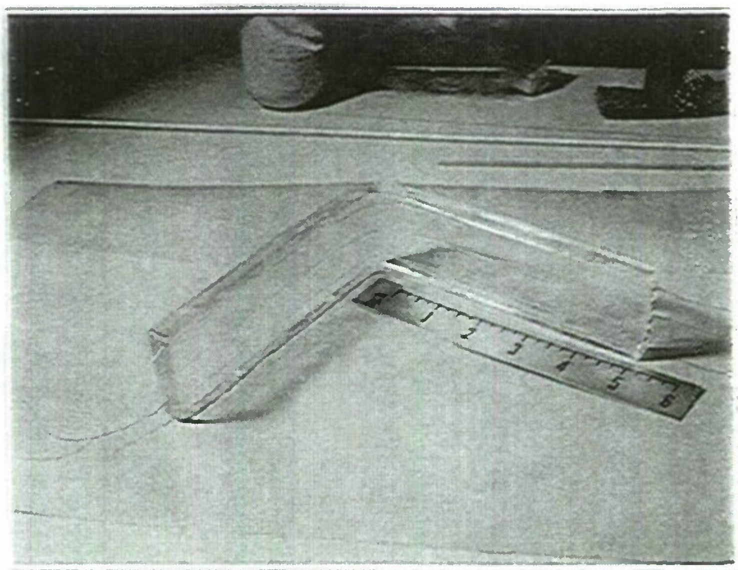
Figure 5-26. Prototype generic part with three embedded AWG and representative of a section of a composite tank crew capsule



Top View of Teflon Mold for Generic Part Showing Three AWG in Position Before Pouring of Resin



View of Teflon Mold with Side Removed and Showing the Three AWG Held in Four Positioning Inserts



View of Generic Part (G2) After Removal from Mold. Shell 815 Clear Resin with Three Embedded AWG of 10 mil Nichrome.

Figure 5-27. Pictures of Teflon Mold and an Actual Prototype Generic Part (G2) Representative of a Section of a Composite Tank Crew Capsule, after Removal from the Mold

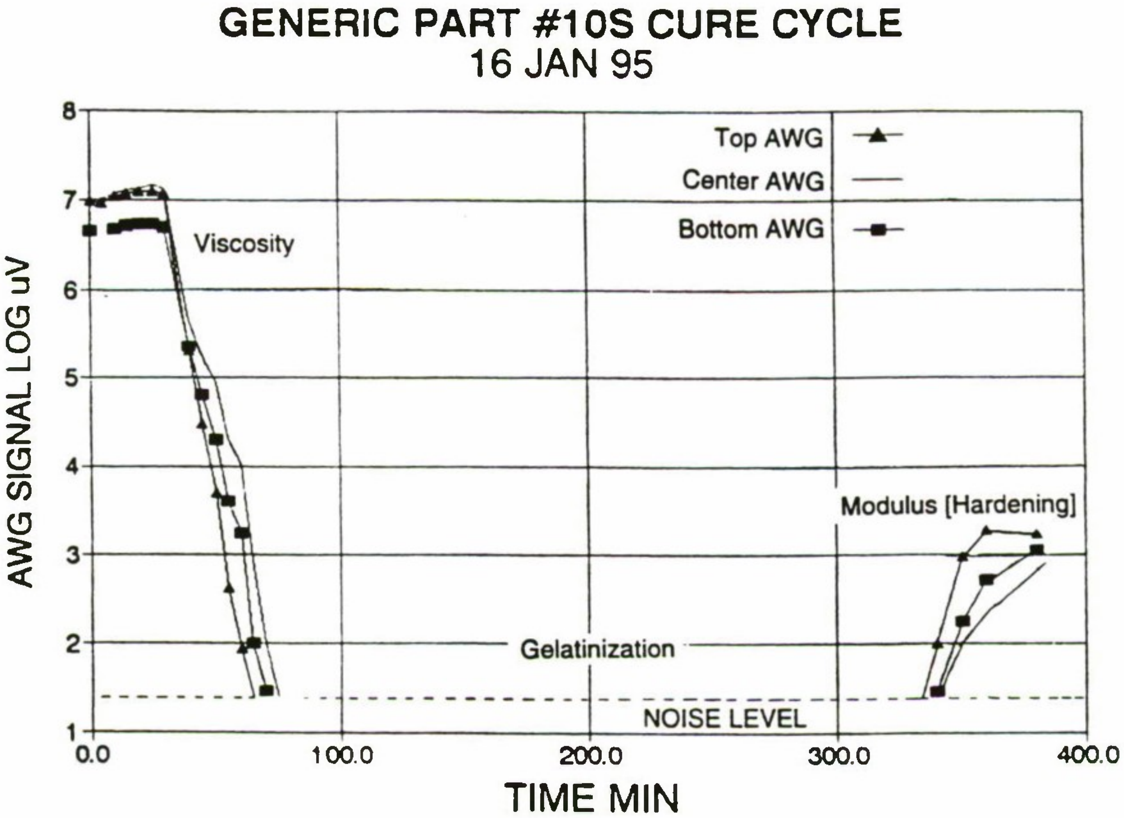
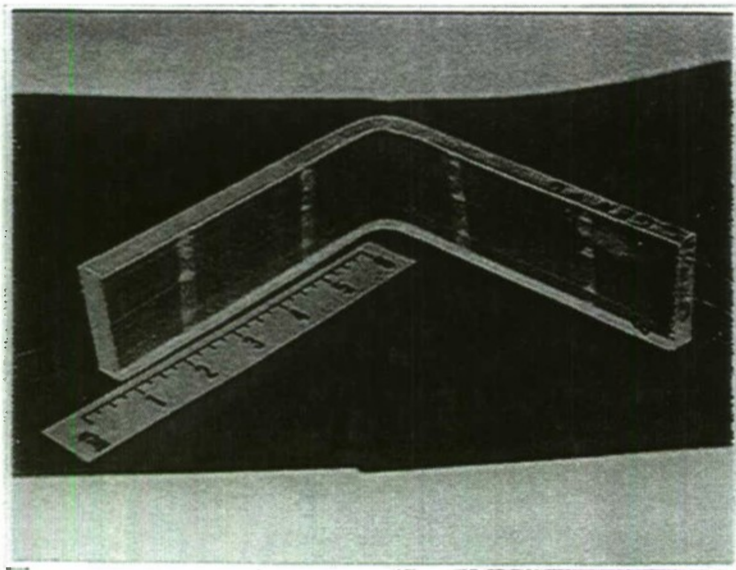
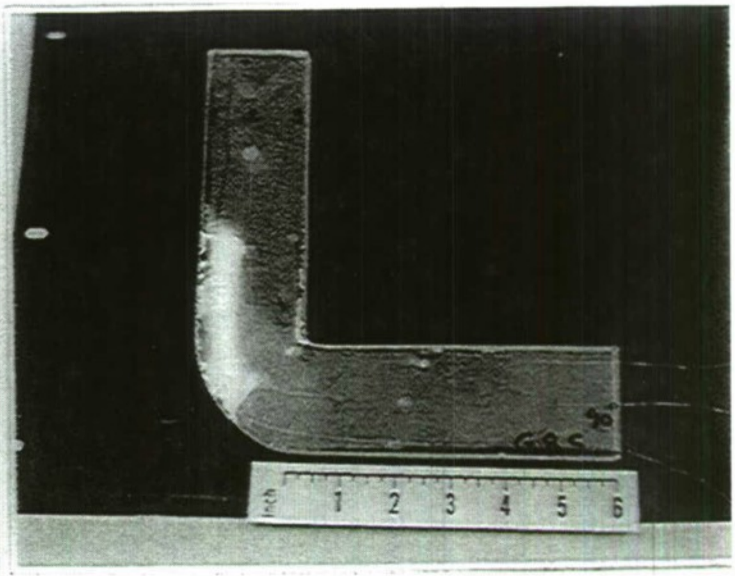


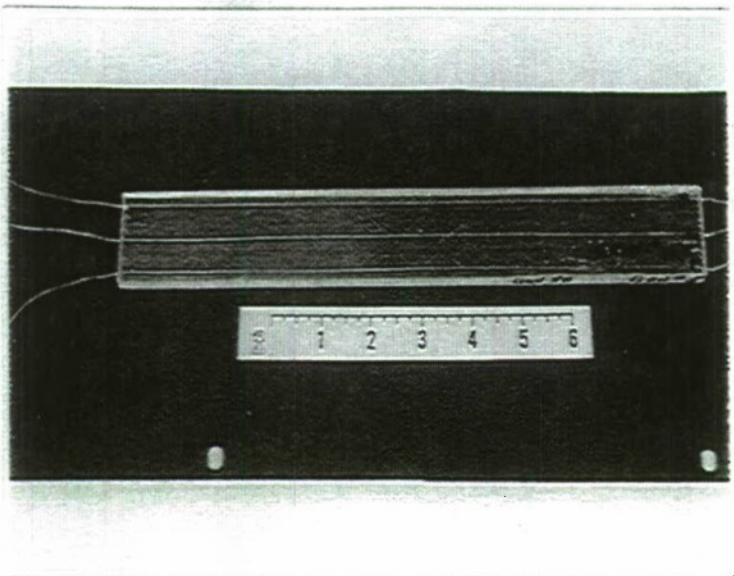
Figure 5-28. Representative cure curves for generic part of clear Shell 815 resin with three embedded 10 mil diameter Nichrome AWG (flat part)



Standard generic part G6, molded from clear Shell 815 resin with three embedded AWG of 10 mil Nichrome



Flat, curved generic part, G8S90°, molded from clear Shell 815 resin



Flat, straight generic part, G5S, molded from clear Shell 815 resin

Figure 5-29. Pictures of generic parts, standard; flat curved and flat straight used for acoustic wave velocity measurements

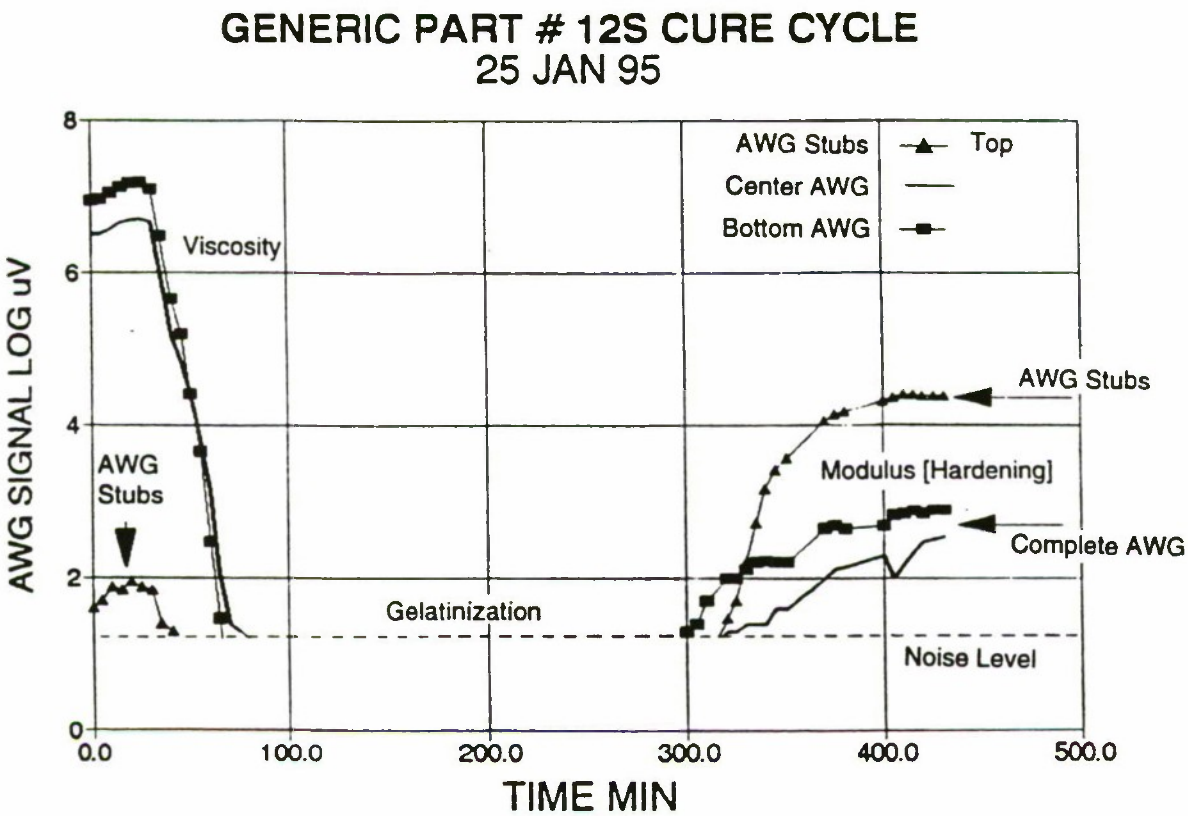


Figure 5-30. Simultaneous room temperature plus infra-red heat acoustic waveguide cure curves for flat-straight generic part G12S. Two complete AWG; two AWG stubs

Table 5-8
Acoustic Wave Velocities Within AWG Embedded Within Generic Parts

GENERIC PART IDENTIFICATION	PROCESSING	WAVE VELOCITIES WITHIN AWG TOP	CENTER	BOTTOM	VELOCITY IN RESIN
G1. Standard	Room Temp.	1000 m/sec	---	1100 m/sec	1200 m/sec
G2. Standard	Room Temp.	1200	---	1070	1160
G3. Standard	5 Hours Heat	1170	950	1100	1270
G4. Standard	Room Temp.	1300	1000	1160	1270
G5S. Flat Straight	Room Temp.	1170	1450	1450	1870
G6. Standard	Room Temp.	1160	1030	1200	1180
G7. Flat Straight	Broken on removal from mold.	No measurements			
G8S90°. Flat Curved	Room Temp.	1290	940	1110	1570

through the curing part) gives little viscosity data but excellent modulus data near the end of the cure.

In Table 5-8, list of generic parts and acoustic wave velocities, it should be noted that the higher the acoustic wave velocity within an embedded AWG, the higher the material modulus. See Section 5.4.7.

5.6 Acoustic Waveguide Sensing of Gas and Bubbles in Curing Resin

In order to assess the performance of embedded acoustic waveguides (AWG) for monitoring the quantity of resin within a liquid composite mold, and also the formation or presence of gas bubbles, some laboratory experiments were carried out. During these experiments some information on the degree of mold filling was also obtained from the AWG signal reduction as molds were filled with resin.

5.6.1 Mold Filling

Mold filling experiments were carried out using an aluminum mold similar to that shown earlier in Figure 5-3, but with a transparent polycarbonate face so that the resin flow could be observed. In addition, resin was poured into one end of the mold during filling and the central resin pouring region was eliminated. As resin was poured into one end of the mold with a transparent face, Figure 5-6, it was observed that the resin flowed readily through the mold and around the glass fibers apparently without the formation of gas bubbles or voids. It took some time, ~16 minutes, to fill this small model mold completely, as the resin was gravity fed. In addition, as the resin gradually filled the first leg of the mold, the AWG signal would gradually reduce in magnitude, then remain steady, until the bottom part of the mold filled sufficiently with resin to cover the AWG in that location. The resin flow rate was reducing at this time and vacuum application was required to pull the resin up the final leg of the mold. This vacuum application caused the release of trapped gas bubbles and these were observed for several seconds emerging from the mold lower surface and traveling to the resin surface at the final leg of the mold. The AWG signal levels recorded during the filling of the mold with resin are shown in Figure 5-31. Although this type of AWG measurement of mold filling is not sensitive enough to detect the presence of trapped gas bubbles, it does provide a form of measurement which could be useful for repeated use of a particular mold. With large size practical molds, several AWG would most likely be required.

A mold filling curve, Figure 5-45, was also obtained during the filling of the generic part mold with resin, Section 5.11. This curve is a plot of the signal transmission changes for a single 20 mil diameter Nichrome AWG passing through the mold containing 40% by weight of "E" glass fibers.

5.6.2 Gas Bubbles

When the gas bubbles were observed during mold filling, the embedded AWG was also used in the listening mode (~3 kHz to ~80 kHz) to determine whether the generation and flow of bubbles to the resin surface caused any acoustic emissions, as is the case for less viscous liquids[2]. No acoustic emissions were sensed, and it is believed this is because of the cushioning and sound dampening properties of the viscous resin.

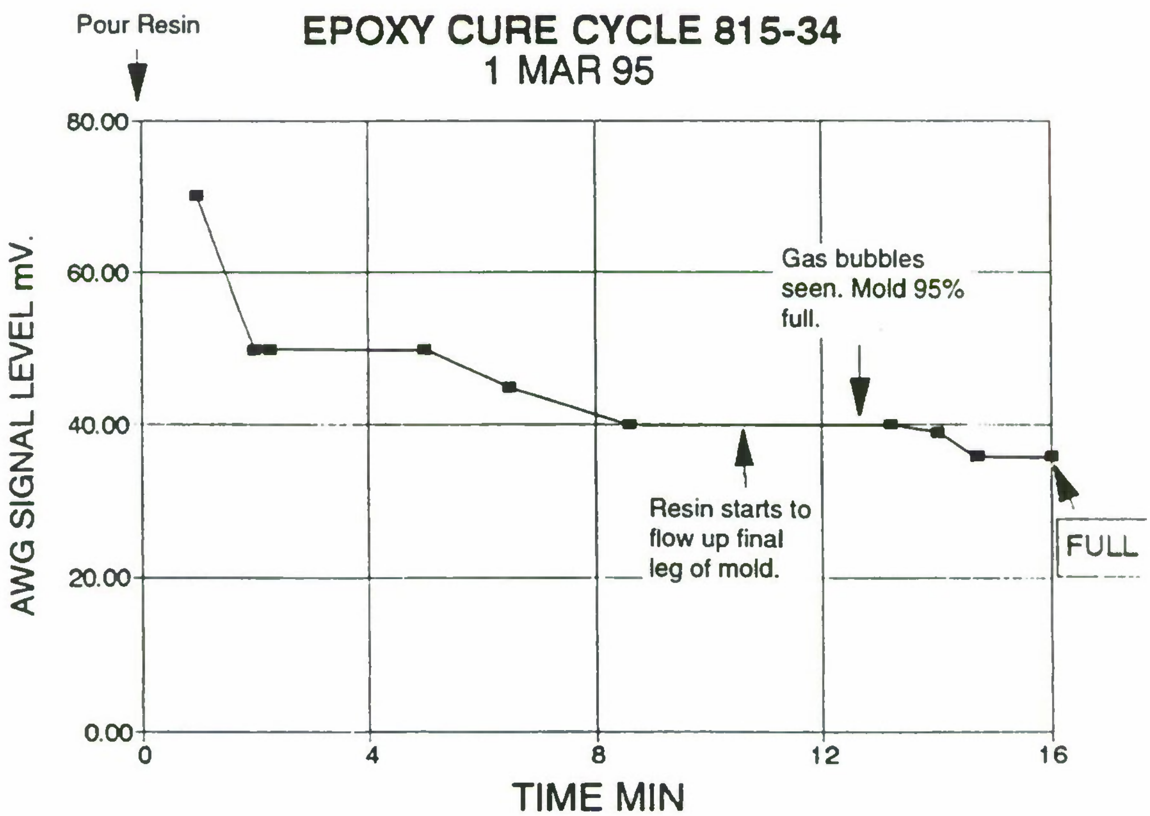


Figure 5-31. AWG signal levels recorded during filling of the mold with resin

5.6.3 Gas Content of Resin

In an attempt to determine whether Shell 815 resin when mixed with hardener and ready for pouring into a mold contains trapped gas, simultaneous AWG cure curves were obtained using normally mixed and "degassed" resin. Both resin samples were mixed at the same time and one sample was placed under a partial (<50 Torr) vacuum for 10 minutes. The molds used for cure monitoring were aluminum containers holding about 200 cm^3 of resin each, and the simultaneous AWG cure curves are shown in Figure 5-32. These curves are very similar, especially in the time after resin pouring and when the viscosity is increasing, also the gelation phase time and changes. The hardening phase parts of the cure curves are also similar, with the non-degassed resin curve indicating a higher signal level and presumably a slightly higher modulus. From this experiment it can be concluded that degassing this type of resin before pouring has no obvious effect on the cure curve. For example, if the gas content of the resin samples was substantially different after pouring, then due to acoustic wave attenuation and bubble dispersion, the initial part of the two cure curves up to gelation would be anticipated to be different. It should also be noted that at the maximum exotherm temperature rise near 88 minutes, the shape of the two cure curves is very similar. This is another indication of a similar gas content for each resin sample.

In order to gather information in regard to the gas content of resin, a series of experiments were carried out using the apparatus shown in Figure 5-33. This system allowed AWG monitoring both along an AWG as usual, and also between two AWG spaced ~2 cm. It was felt that monitoring between AWG would be more sensitive to the gas content of resin. The resin was saturated with nitrogen gas using a bubbling method first used several years ago[7] in mineral oil. Results for mineral oil and repeated in this study are shown in Figure 5-34. Basically, nitrogen gas is bubbled into the mineral oil via a perforated submerged pipe and the variation in the acoustic signal level transmitted between immersed AWG is recorded.

As can be seen in Figure 5-34 for mineral oil, after gas bubbling for about 30 minutes, the AWG signal level reduces by an order of magnitude. At 60 minutes, the bubbling is turned off, no bubbles are visible, yet the AWG signal remains reduced and takes a few minutes to return to its original level. Similar results for Shell 815 resin (without hardener) are shown in Figure 5-35. The main differences are 20 minutes of nitrogen bubbling and over two hours to recover to the normal signal level, all without visible gas bubbles. Long recovery time would be anticipated for the more viscous resin compared with mineral oil. Actual oscillographic pictures of the signals transmitted between AWG for the saturated and non-saturated conditions for both liquids are shown in Figure 5-36.

In order to determine whether signal transmissions between AWG would sense the presence of gas in the resin during the cure cycle, simultaneous AWG cure curves were obtained for Shell 815 resin for signal transmission along an AWG and between AWG, Figure 5-37. Approximately 10 minutes after pouring of the resin the nitrogen bubbling within the resin was activated for a 10 minute time period. As can be seen in Figure 5-37, the gas saturation condition was sensed by the order of magnitude reduction in the acoustic signal level transmitted between AWG, but not by the signal being transmitted along one AWG. Furthermore, the reduced signal level sensed between AWG remained when the nitrogen bubbling ceased, and only returned to the apparent normal level about 40 minutes after pouring of the resin. This experimental result provides convincing evidence that the monitoring of acoustic signal levels transmitted between immersed AWG during the resin cure cycle can yield information on the gas content of the resin. In addition, acoustic waveguide transit times (velocity) both between AWG and along an AWG were recorded during this experiment, Figure 5-38. As might be expected under gas saturation conditions, the wave transit time between AWG slowed up, with a change of transit time of ~90 μsec (in the resin) to ~180 μsec (in the gas). This can be roughly interpreted as 90 μsec wave

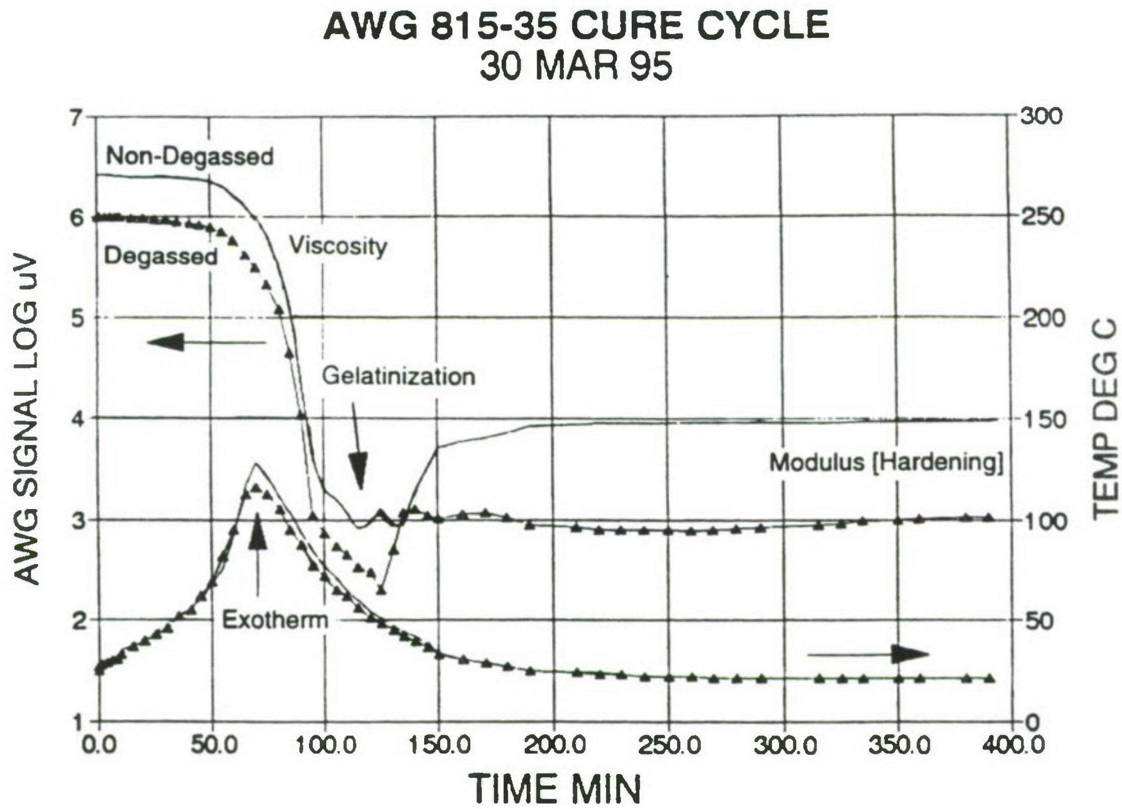


Figure 5-32. Simultaneous AWG cure curves for Shell 815 resin cured at room temperature using both degassed and non-degassed resin (0 to 350 minutes)

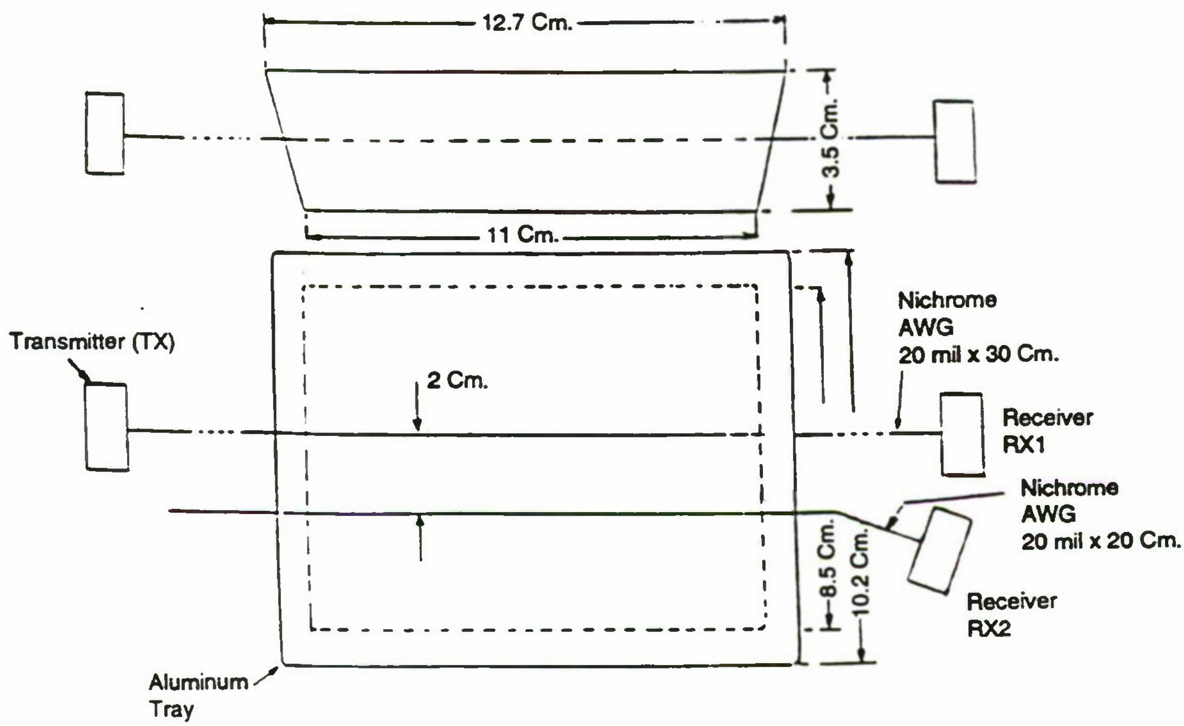


Figure 5-33. Arrangement for the acoustic waveguide cure monitoring of resin by measuring the signal attenuation along one waveguide and between two waveguides

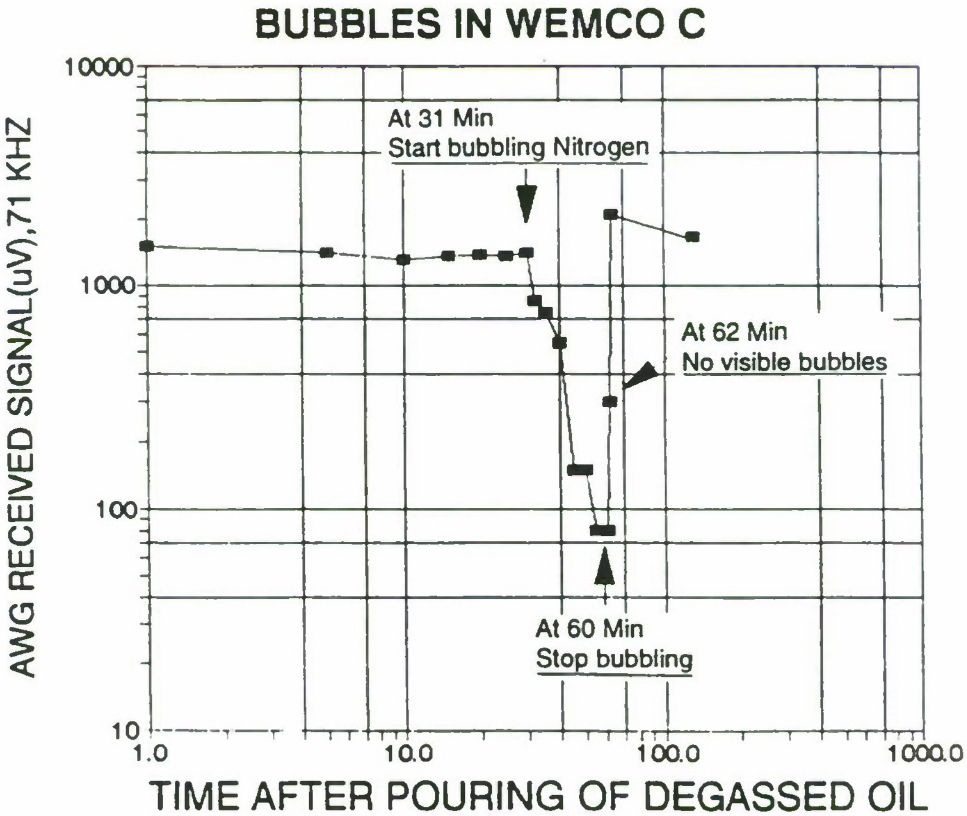


Figure 5-34. The influence of the gas content of mineral oil on the attenuation of ultrasound transmitted between immersed AWG

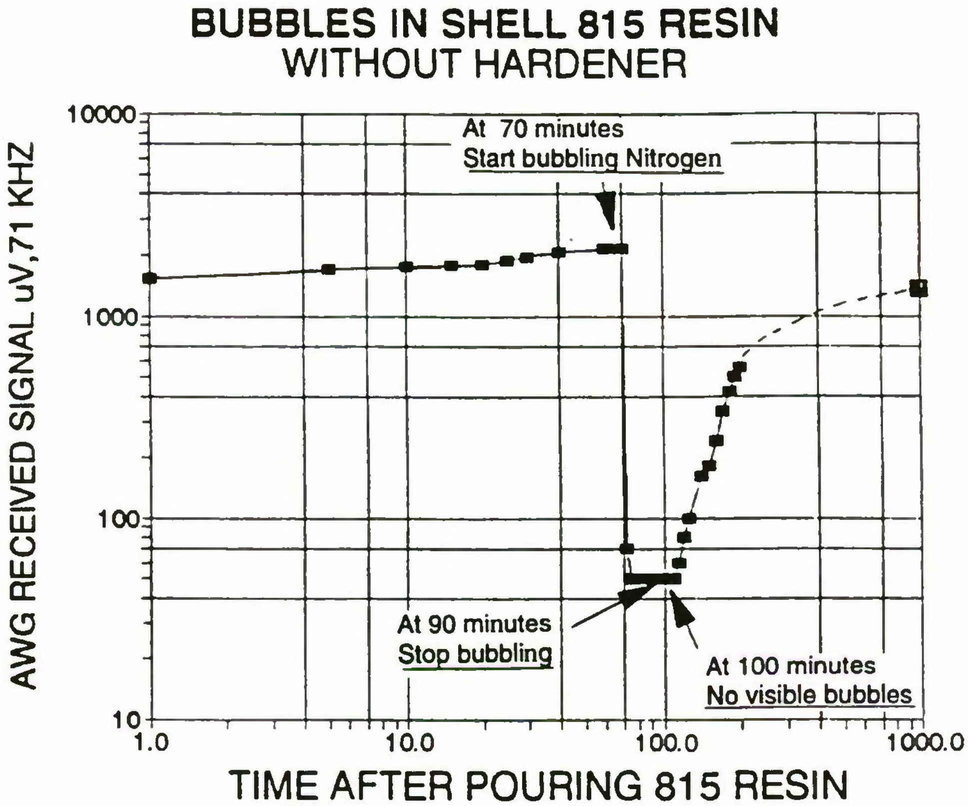
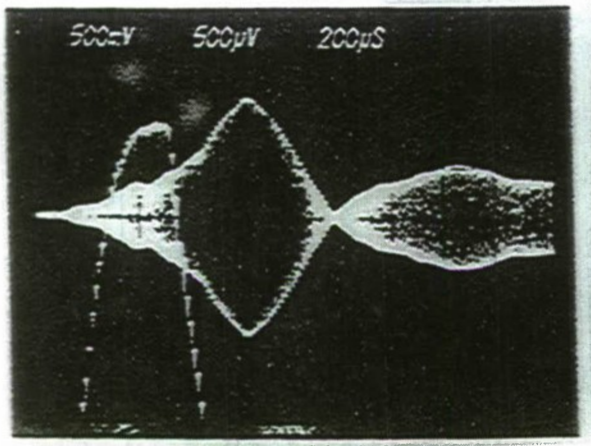
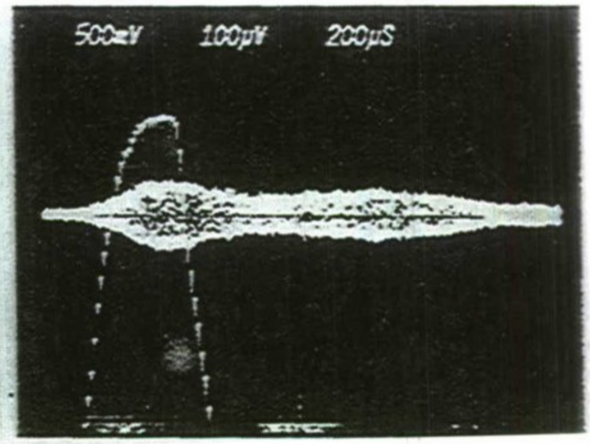


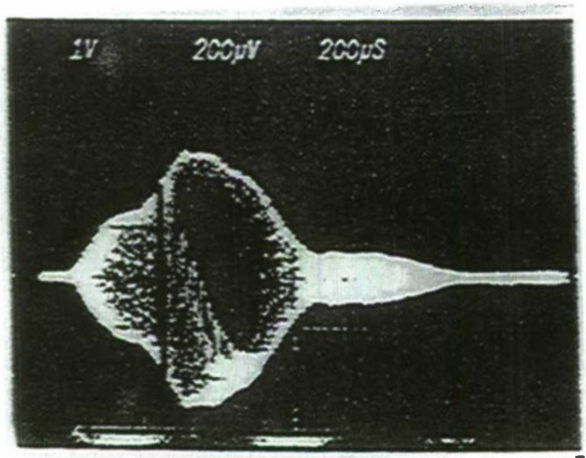
Figure 5-35. The influence of the gas content of Shell 815 resin on the attenuation of ultrasound transmitted between immersed AWG



AWG signal for mineral oil at 64 minutes, 25 seconds, no visible bubbles. Signal level ~1200μV and returning toward original 1500μV level.



AWG signal for mineral oil at 60 minutes, 20 seconds, no visible bubbles. Saturated condition. ~70μV.



AWG signal for Shell 815 resin at 185 minutes, no visible bubbles. Signal level 500 μV and returning toward original 1500 μV level.

AWG signal for Shell 815 resin at 100 minutes, no visible bubbles. Saturated condition, ~50 μV

Figure 5-36. Attenuation of signals transmitted between AWG immersed in both mineral oil and Shell 815 resin when saturated with nitrogen gas

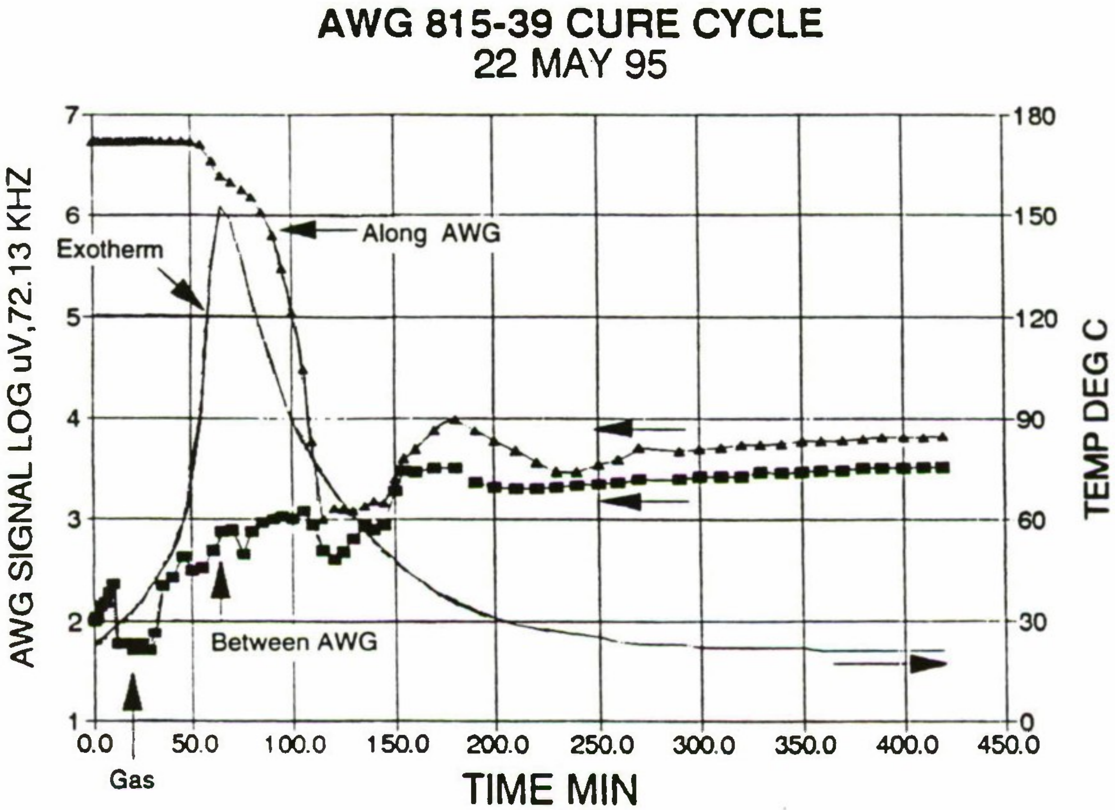


Figure 5-37. Embedded AWG cure curves for Shell 815 resin Specimen #39. AWG transmission along and between AWG. Gas bubbles in resin sensed by transmission between AWG

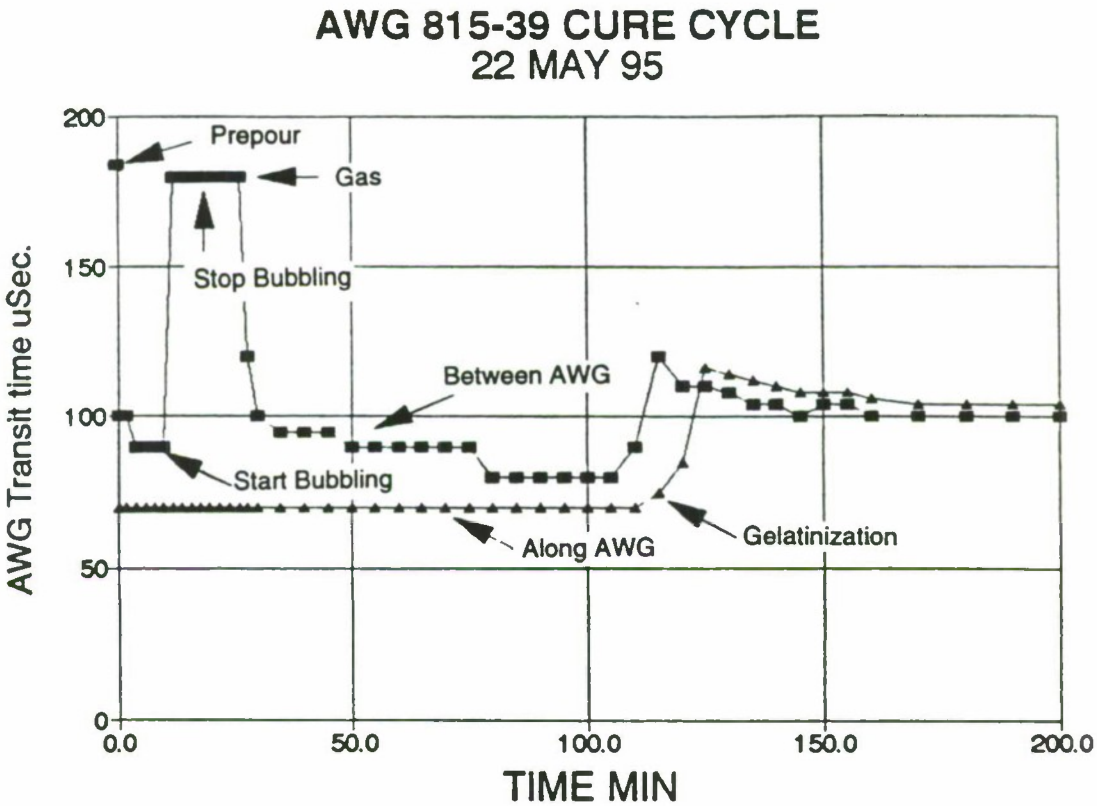


Figure 5-38. AWG transit times (velocity) along AWG and between AWG versus cure time for Shell 815 resin Specimen #39. Transit time changes between AWG indicate resin gas content

travel time for 2 cm between AWG, or ~220 m/sec, which is of the order of that expected for nitrogen gas; i.e., 330 m/sec. Transmission velocities within curing resin are in the ~1000 to ~2000 m/sec range.

5.7 Acoustic Waveguide Measurements of Resin Viscosity

During the early part of the curing cycle of resin the viscosity increases rapidly and typically increases several orders of magnitude until gelatinization (the transition from a liquid state to that of a rubbery gel). An embedded AWG will track this viscosity increase via several orders of magnitude signal decrease. In order to calibrate the AWG sensor for measuring viscosity, measurements were made in castor oil taken to -49°C. Castor oil was used as it has large and known viscosity changes with falling temperatures. These measurements and procedures are covered in detail in Appendix C.

In Table 5-9 some AWG viscosity measurements for different materials are listed.

Table 5-9
Estimated Viscosity Values at Gelation

MATERIAL:	VISCOSITY (Poise)
Jello	134
Polyurethane	2×10^5
Shell 815 Resin	10^6 to 3×10^7
Concrete	3×10^9
Polymer Concrete	10^{11}

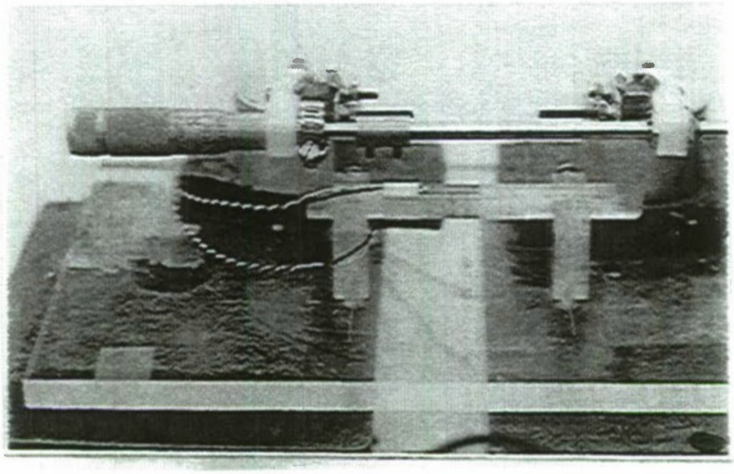
5.8 Acoustic Waveguide Sensing of Strain Inside Resin

The values of strain inside materials are usually estimated from surface strain gauge measurements based on material uniformity. With transparent parts, strain can be measured optically, but as most resins and composites are far from uniform in cross-sectional density and usually opaque, then internal strain values under load are unknown.

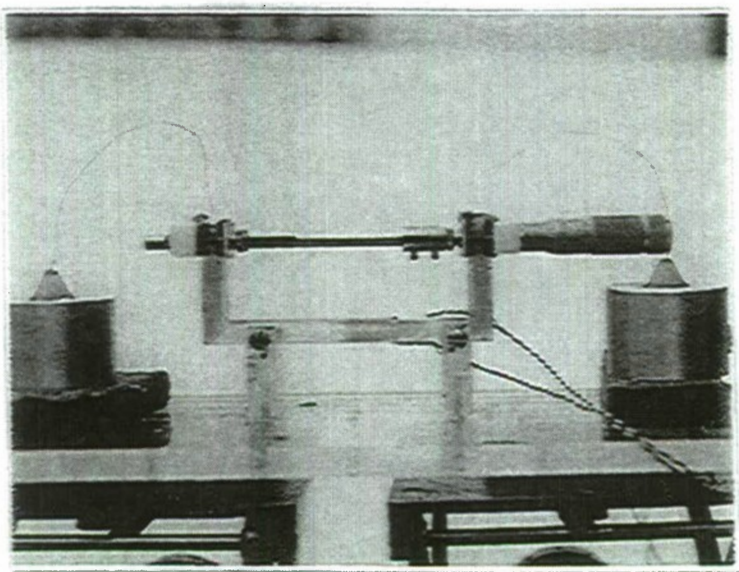
Although all the theoretical aspects are not understood, embedded AWG sensors exhibit changing signal levels under load which correlate with surface strain gauge readings.

Acoustic waveguide strain measuring experiments were carried out using clear resin specimens which were 10 cm wide with a 1 cm x 1 cm cross section and with two right-angled bends and strained using the apparatus illustrated in Figure 5-39. Conventional strain gauges were bonded to either the top or bottom surfaces of the horizontal part of the specimens. Three specimens were examined, each with an embedded AWG in a different location within the horizontal part. The locations chosen were ~1mm from the top surface of the resin specimen, ~1mm from the bottom surface, and in the middle or neutral zone (strain-free when the specimen is strained). In order to introduce strain within a specimen it was only necessary to slightly push the two vertical arms of a specimen toward one another or slightly pull them apart. Precise control of strain within a specimen was achieved using a clamping arrangement and micrometer, as shown in Figure 5-39. For example, about 120 mil of movement (i.e. pushing the specimen vertical arms toward each other, or movement in the opposite direction) would yield a surface strain reading of approximately 2000 compressive or tensile microstrain.

This was the procedure used to strain the three specimens, one with an embedded AWG near the top surface, one with the embedded AWG in the neutral zone, and one with the embedded



VIEW SHOWING RESIN SPECIMEN
AND SURFACE STRAIN GAUGE WITH
MICROMETER FOR ADJUSTING STRAIN



VIEW SHOWING STRAINED RESIN
SPECIMEN AND AWG BONDED TO
ACOUSTIC TRANSMITTER AND RECEIVER

Figure 5-39. Pictures showing strained clear resin specimen with embedded AWG, surface strain gauges and acoustic transmitter and receiver

AWG near the bottom surface. For each specimen, as it was strained to ~2000 microstrain in both tension and compression (as measured by surface mounted conventional strain gauges), measurements were made of the magnitude of the AWG transmitted signal, Figure 5-40. These curves show that for the AWG close to the top surface of a specimen, the AWG transmitted signal falls under compression and increases under tension. While for the AWG embedded near the bottom surface, the AWG signal changes are reversed. With the AWG in the neutral or strain free zone, only small changes in the AWG transmitted signal occur under both compression and tension. It should be emphasized that these measurements indicate typical signal trends which are repeatable. However, actual values can change on repeated testing, i.e., if the transmitted signal resonant frequency changes, or the AWG is bent at entry or exit which changes the terminal signal transfer function.

In order to yield more information, optical strain patterns were also measured in a clear resin specimen strained to 2000 microstrain in both compression and tension, Figure 5-41. Although these TV screen pictures are not very clear they do show increasing numbers of strain fringes with increasing strain, and the compression strain patterns appear to be the reverse of the tensile strain patterns.

5.9 Design and Construction of Sonic Meter

As shown earlier in Section 5.4.1, Figure 5-3, an oscilloscope is used for measuring the peak value of the acoustic signal transmitted through an embedded AWG, and the AWG cure curve is plotted from these data. In order to provide a real time cure curve during liquid composite molding the cure monitoring instrumentation package delivered to TARDEC will not include an oscilloscope. Instead of using an oscilloscope and recording and hand plotting of data, the 60-80 kHz signal levels at the AWG receiver will pass through a 30 kHz high pass filter (to remove background noise) and be fed to a special logarithmic, highly sensitive analog meter, which can measure signal levels from ~10 microvolts to ~1 volt on a single scale. The output from this analog meter will then be fed to a programmable X-Y recorder for direct real-time display of the AWG cure curve.

This analog meter, designed and constructed at Westinghouse Science & Technology Center is shown in Figure 5-42.

Details of the Sonic Meter:

The sonic corona meter (Figure 5-42) is a logarithmic peak reading device that will respond to the largest repeated acoustic emission pulse per power frequency cycle. A block diagram of the device (Figure 5-43) illustrates the wide dynamic range, low noise input preamplifier that responds to signals ranging from ~10 μV to about ~1 V in magnitude, for a dynamic range of 10^5 or 100 dB. The preamplifier bandwidth is from 10 Hz to 600 kHz. The next stage is a logarithmic response detector amplifier with a bandwidth of 5 kHz to 100 kHz. These stages drive a peak detector and meter circuit to provide a meter range from 2 to $2x10^5$ units on a single meter scale. When the sonic meter is used in noisy environments, a 30 kHz high pass filter is added.

5.10 Instrumentation for Acoustic Waveguide Cure Monitoring

The instrumentation for acoustic waveguide cure monitoring which is to be sent to TARDEC is shown in Figure 5-44. This instrumentation package consists of a programmable Wavetech, Model 80, 50 MHz pulse function generator, an analog sonic meter (see 5.9), and a

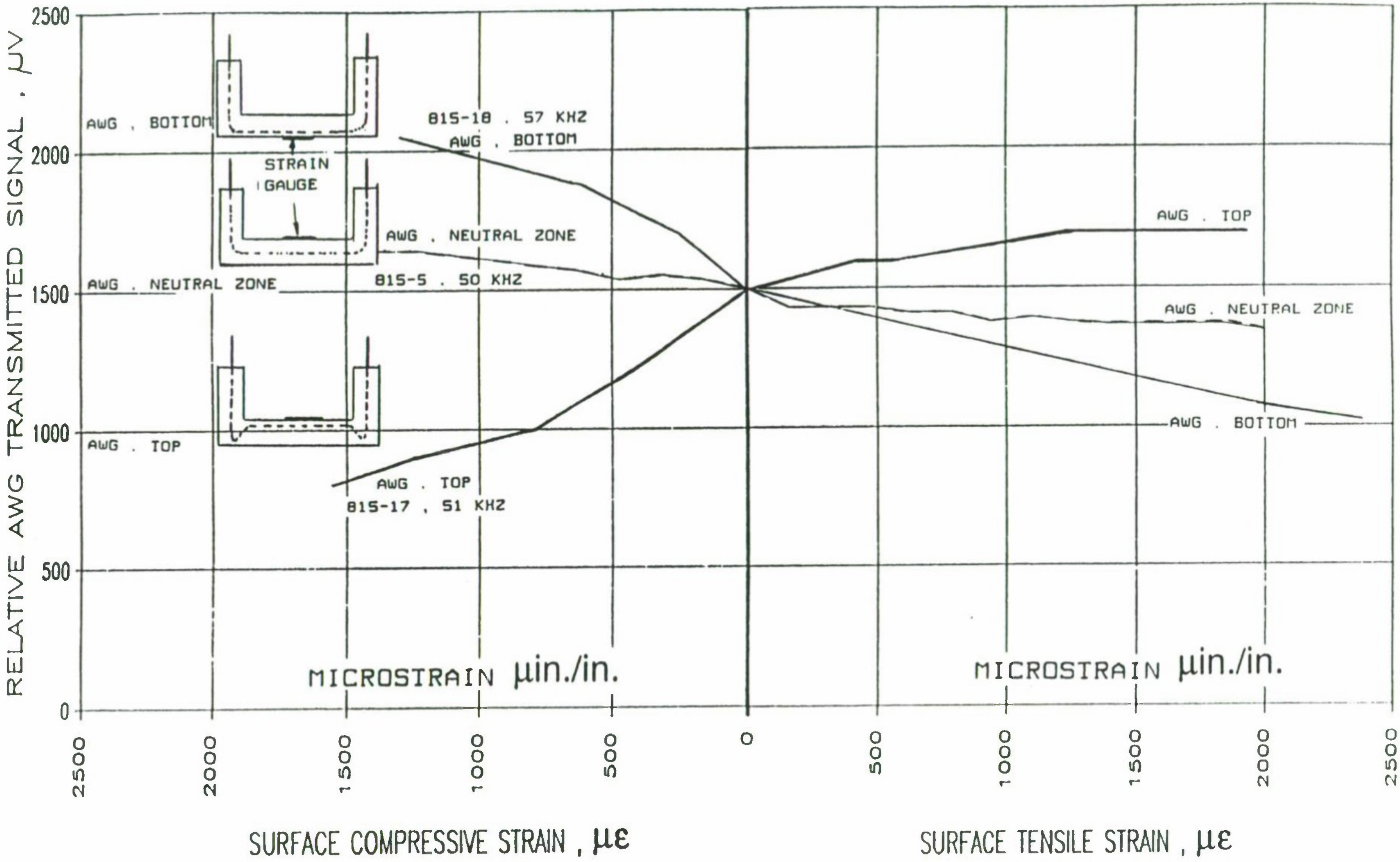


Figure 5-40. Comparison of relative embedded AWG transmitted signal levels and surface strain gauge readings for resin parts strained in both compression and tension

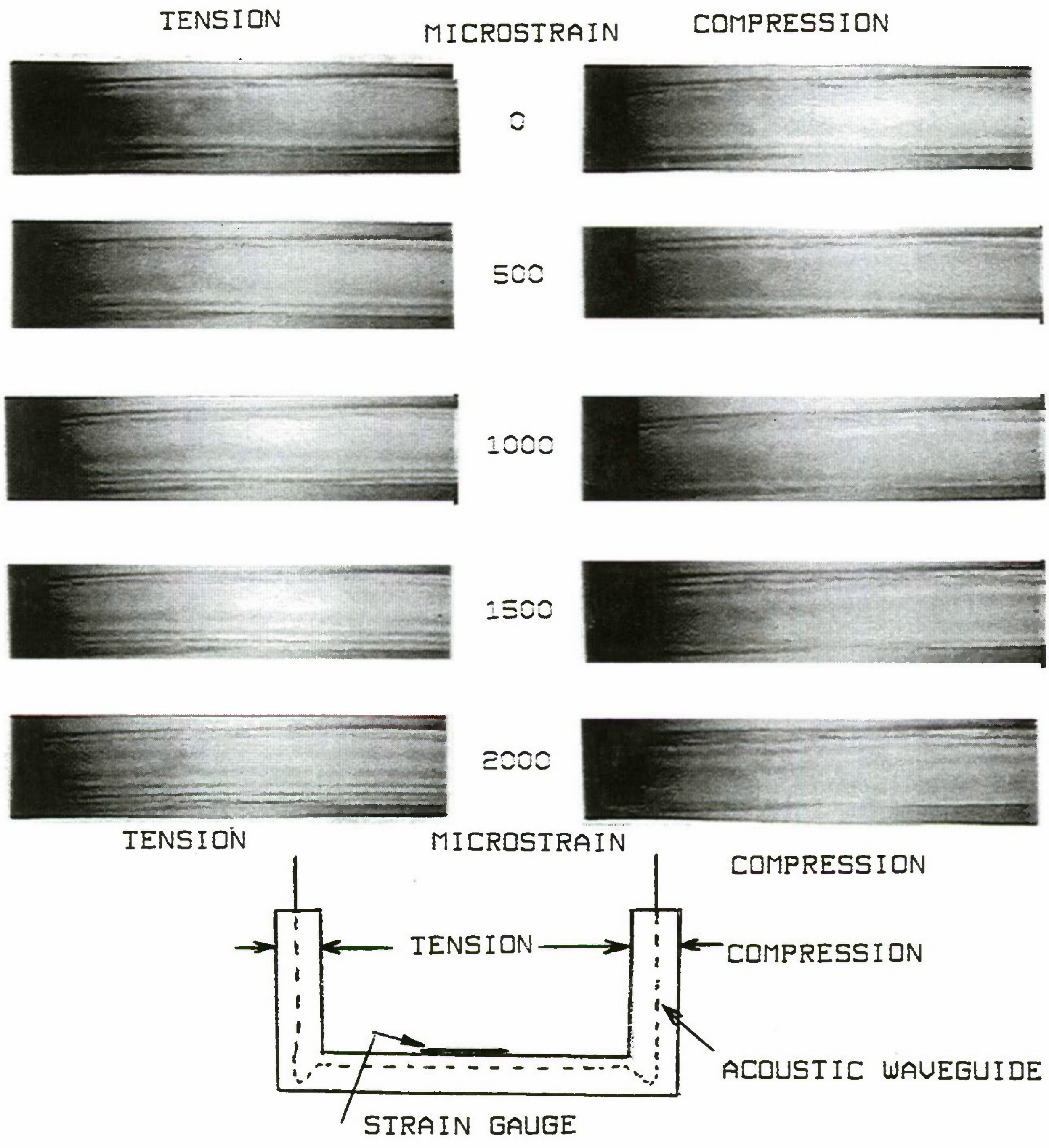
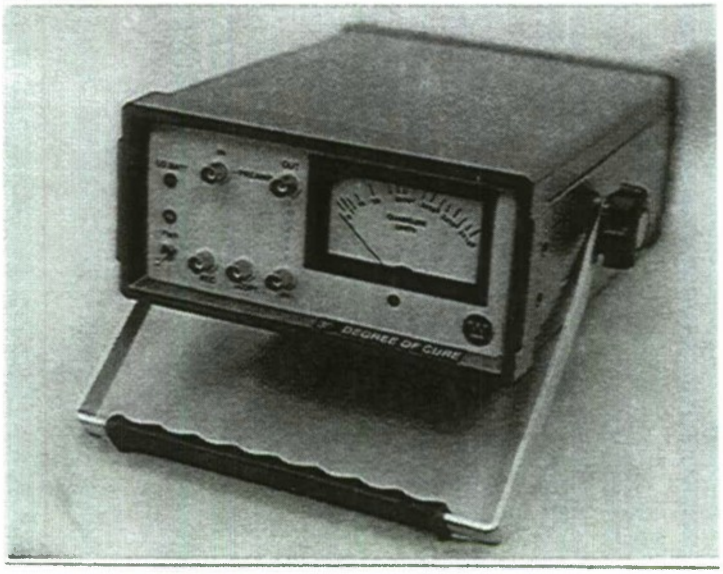


Figure 5-41. Optical strain patterns within clear resin specimen stressed to 2000 surface microstrain in both compression and tension



Type of sonic meter under construction

Figure 5-42. Westinghouse logarithmic response peak reading sonic meter

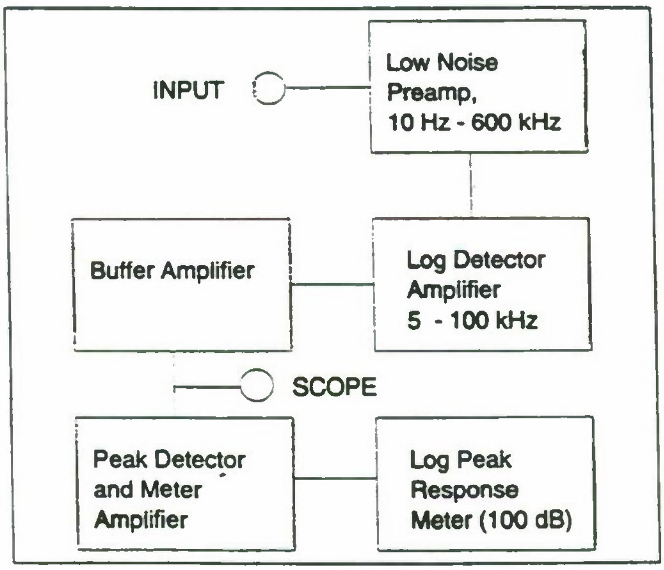
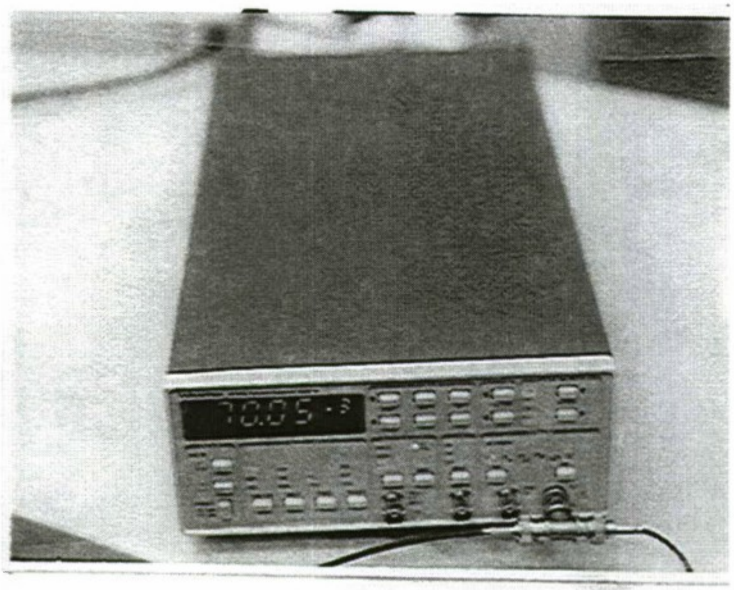
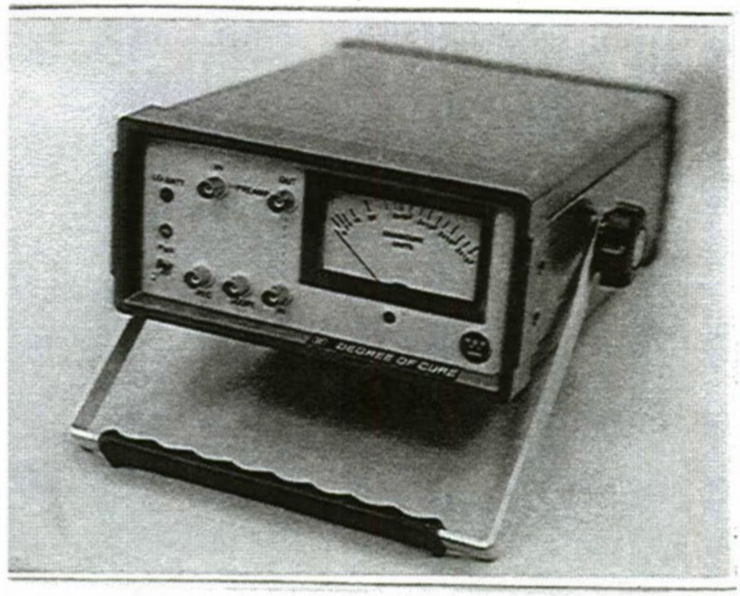


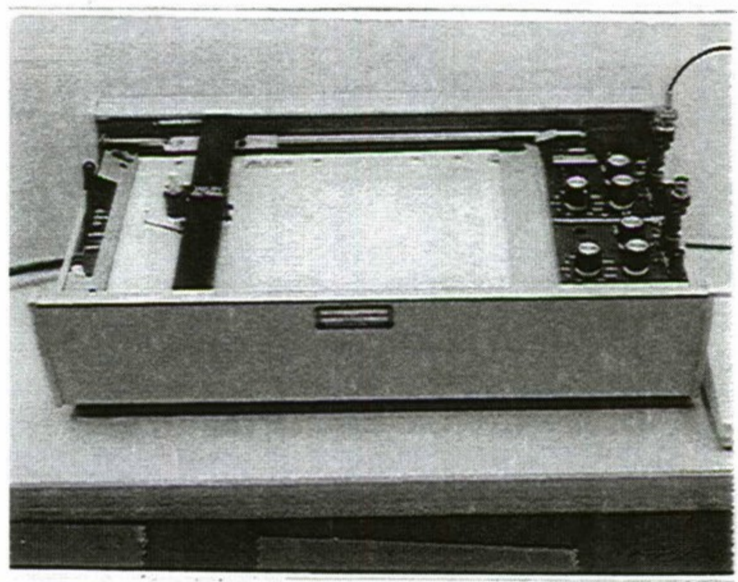
Figure 5-43. Block diagram of the Westinghouse logarithmic response peak reading sonic meter



Programmable Wavetech, Model 80, 50 Mhz
Pulse Function Generator



Type of sonic meter under construction



Programmable Allen 7000 Series X-Y Recorder

Figure 5-44. Pictures of instruments to be used for cure monitoring (TARDEC)

programmable Allen 7000 series X-Y recorder. Also included in the package are a 30 kHz high pass filter, 60-80 kHz acoustic transmitter/receivers and 3 meters length each of both 10 mil and 20 mil diameter Nichrome acoustic waveguide.

5.11 Acoustic Waveguide Cure Monitoring Demonstration Video

In order to save time and costs, instead of giving an AWG cure monitoring demonstration at TARDEC, an instructional video of this operation was made. In this video, the procedure for assembling the AWG system within the generic part mold is seen, as well as the separate instruments and their function. Also, the AWG cure monitoring of a generic part made from Shell 815 resin with 40% by weight of "E" glass fibers is shown, beginning with the mold filling with resin. The mold filling X-Y plot is seen in Figure 5-45 and also the X-Y plot of the complete cure curve, Figure 5-46. The cure monitoring demonstration video was delivered to TARDEC on November 8th, 1995. A picture of the generic part mold and cure monitoring instrumentation is shown in Figure 5-47. This shows the instrumentation to be delivered to TARDEC. The pulse signal generator shown is the latest programmable model, while the signal generator seen in the demonstration video is an earlier model.

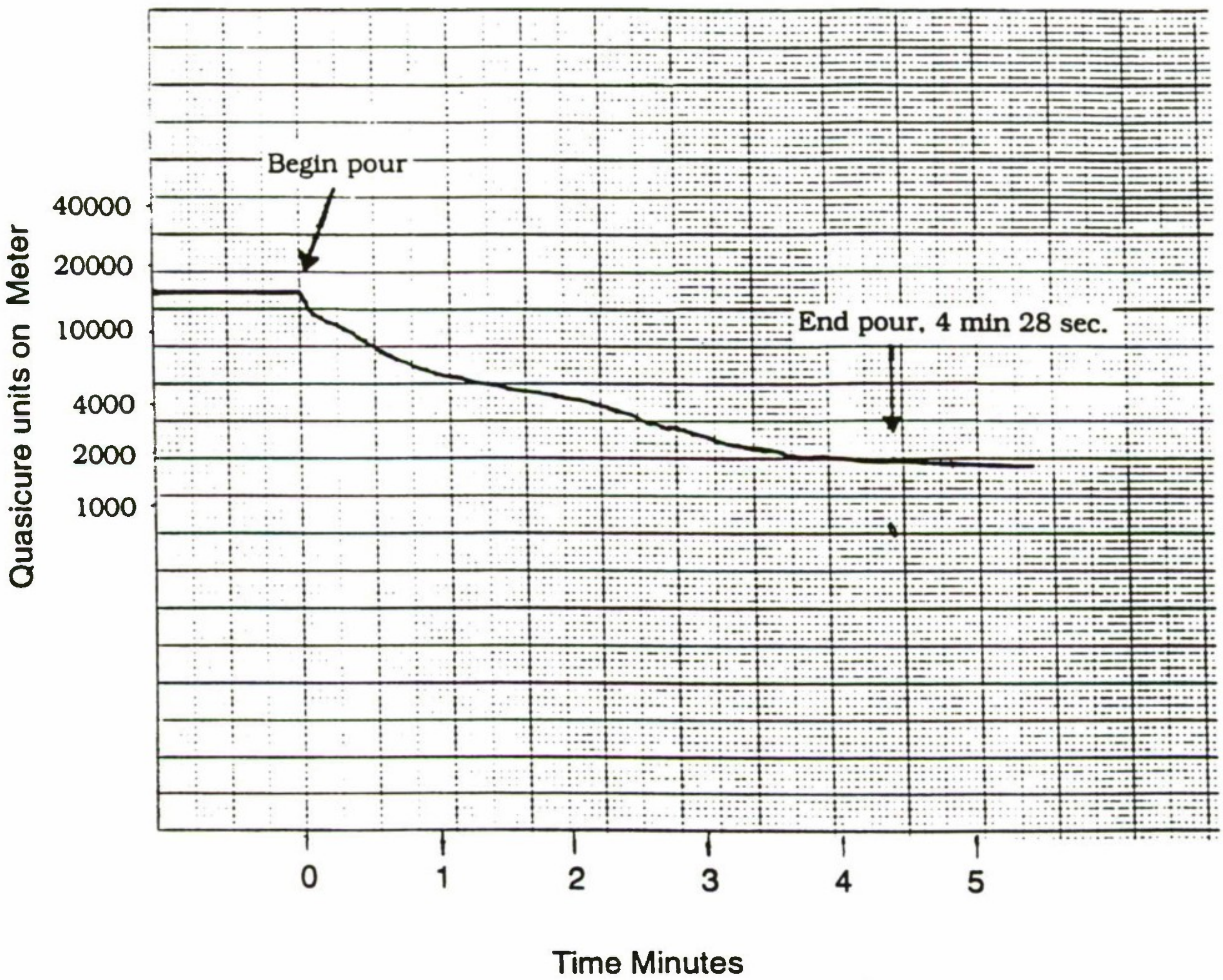


Figure 5-45. AWG signal levels recorded during the filling of the generic part mold with resin. Mold containing 40% by weight of "E" glass fibers and one 20 mil diameter Nichrome AWG.

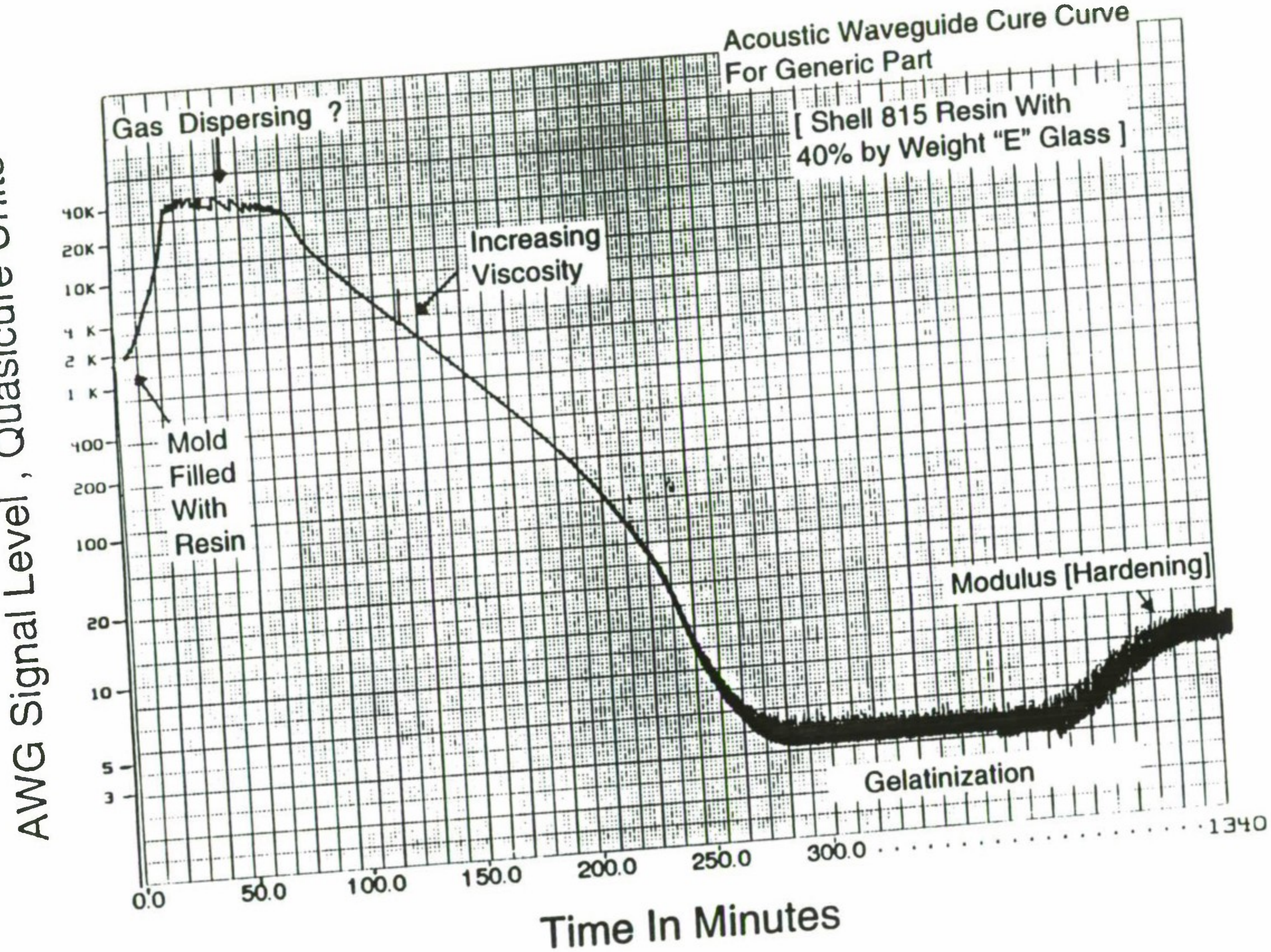


Figure 5-46. Acoustic waveguide cure curve for generic part(shown in demonstration video).

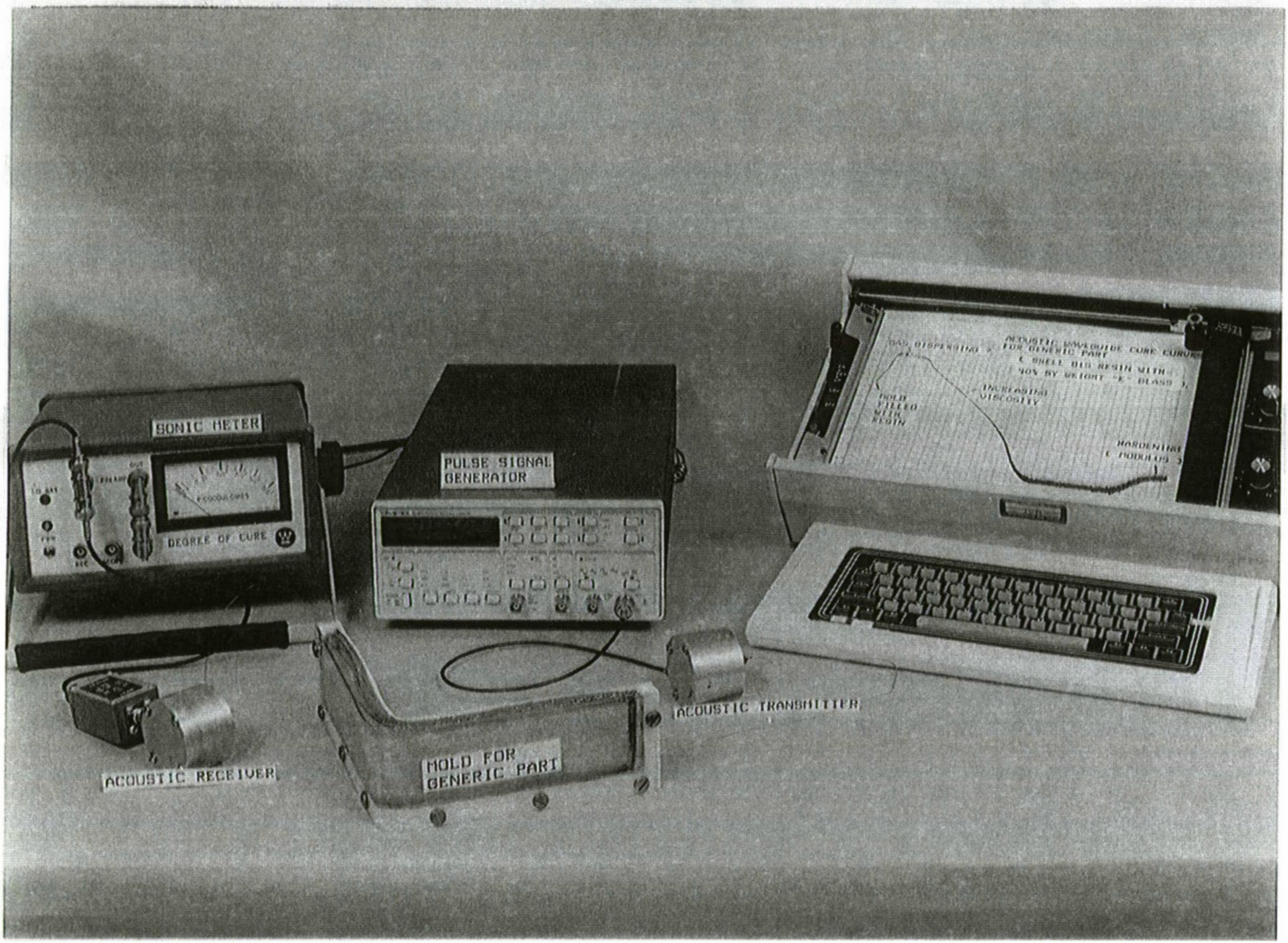


Figure 5-47. Picture showing the generic part mold and cure monitoring instrumentation.

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APPENDIX A
Recommended Follow-On Program

Having now established a firm basis and a better understanding of AWG composite process monitoring and N.D.E. applications, the need now is to transition this technology to an actual resin transfer molding (R.T.M.) machine and practical size composite parts. The outline for a transition program, the issues involved and tasks is given below:

Suggested New Program: Transition of AWG Laboratory Cure Monitoring and N.D.E. Techniques to Actual R.T.M. Machine and Practical Size Parts:

Issues and Problems to be Addressed

- Similar small size R.T.M. at both TARDEC and (W) STC.
- Common Composite part for both composite armored vehicle (CAV) and automobile industry.
- AWG entry/exit R.T.M. machine.
- Acoustic transmitter/receiver locations on R.T.M. machine.
- Background noises from R.T.M. machine and factory environment.
- Locations and securing of AWG within composite part.
- Feedback of AWG signal and control of R.T.M. process.
- Rapid curing of part.
- Mold filling.
- Removal of part from mold and cutting AWG.
- Rejoining or repair of AWG for NDE.
- NDE issues - quality after manufacture - in service health.
- Computerized records to build up data base for this AWG technology application.

These are the issues and problems that need to be addressed although more will probably be thought of later. However, following a program of this nature, with close collaboration between TARDEC and (W) STC, this AWG technology should be ready for factory application by composite parts manufacturers, and also some AWG NDE techniques could be applied to composite vehicles in service.

Suggested AWG Program:

New Program: Transition AWG Process Monitoring and NDE Technology to R.T.M. Machine:

Program Length: 24 months

Phase I - 12 Months - Transition AWG Technology to R.T.M. Machine

Task 1 Purchase or renovate R.T.M. machine - TARDEC/(W) STC installation and operation.

Task 2 Design generic composite part with AWG array. Military/Civilian. (Input from TARDEC/Auto Industry/(W) STC).

Task 3 AWG Entry/Exit R.T.M. machine. Mounting of acoustic transmitters/receivers.

Task 4 AWG cure monitoring in R.T.M. machine both along and between AWG. Data analysis.

Task 5 Exploratory tests on small model parts and molds.

Task 6 Address-background noise problem from R.T.M. machine operation.

Task 7 Cutting and re-joining of AWG. Repair.

Phase II - 12 months - Develop AWG Technology for Control of R.T.M. Process and Factory Application:

Task 1 AWG signal feedback and control of R.T.M. process.

Task 2 Rapid cure monitoring

Task 3 AWG R.T.M. cure monitoring transition to factory - TARDEC/Composite Manufacturers/(W) STC

Task 4 AWG NDE for R.T.M. parts, quality control. Residual strain/modulus.

Task 5 Simplified NDE testing - plug in AWG.

Task 6 Exploratory tests on small model parts and molds.

Task 7 AWG cure monitoring database. Computerized records.

Task 8 In service NDE of composites - TARDEC/Composite Manufacturers/(W) STC.

These phases and tasks represent a type of development program which could help in the application of composite parts to both military and civilian vehicles.

APPENDIX B
Acoustic Waveguide Theory

The basic concept of AWG cure monitoring is that the amount of sound transmitted through an embedded AWG is closely related to the difference between the acoustic impedance of the AWG and the acoustic impedance of, and the pressure exerted by, the host material which surrounds the AWG. The acoustic impedance of the medium which surrounds the AWG depends upon the density of the host material times the velocity of sound through it. Thus, any physical or chemical changes which affect the density of the host material or velocity of sound through it will alter the acoustic impedance and change the amount of sound transmitted through the AWG. Since the density and velocity of sound through a host material are affected by temperature, stress, strain and impact on the host material, all these things can be monitored by means of the AWG. It is even possible to obtain an approximate value for Young's modulus from the information obtained through the AWG. The transit time of the sound wave through the AWG is also affected by these factors and can also be used to monitor changes that occur in the host material.

THEORETICAL ASPECTS

The actual wave propagation modes and velocities along acoustic waveguides can be very complex and were first analyzed by L. Pochhammer[3] in 1876. Since that time a very large number of papers have been written on the subject. Papers by Redwood,[4] 1959, and Thurston,[5] 1978, help in the understanding of guided wave propagation. More recently, studies by Nicholson,[6] 1991, and Nayfeh and Nagy,[7] 1995, yield further insight into acoustic wave propagation in AWG both in air and within a host material.

In this present AWG work on cure monitoring it has been helpful to identify guided wave propagation by tuning the system so that the received acoustic pulse duration time is at least 10 times longer than the transmitted pulse duration time. A condition first recognized by May[8] in 1960. In addition, during AWG cure monitoring the signal changes measured during the viscosity (η) and hardening (modulus (Y)) phases are generally related to the square root expressions below which are a simplification of equations developed by Roth and Rich[9] during their work with an ultrasonic AWG probe for viscosity measurements.

Viscosity - attenuation proportional to $[\rho\ \eta]^{1/2}$ Modulus - attenuation proportional to $[\rho\ Y]^{1/2}$.

where ρ is density.

The basic general concept of acoustic waveguide monitoring is simple, i.e. acoustic signals propagating within a guide will remain confined inside the guide provided there is a large mismatch between the acoustic impedance of the guide material and that of the surrounding medium (solid waveguide in air, for example). However, this premise raises several questions in regard to the influence on the waveguide transmitted signals of such factors as pressure on the waveguide surface (improved acoustic coupling) and the viscosity and temperature of the surrounding medium. Theoretically, it is helpful to consider the expressions and formulae for the velocity and attenuation of acoustic waves in gases, liquids and solids and their relationship with the acoustic impedances of these media. These expressions and formulae are listed below:

- <u>Acoustic Impedance</u> - The characteristic impedance or specific acoustic impedance of a material depends on the product of the density (ρ in kg/m^3) of the material and the velocity (c in m/s) of sound in that material and is expressed as ρc in kg/m^{2}s.

- Transmission of Acoustic Waves at the Interface Between Materials of Different Acoustic Impedance - In order for ultrasound to pass without loss from one medium to another, it is necessary for the characteristic impedance of the media to be identical. Obviously, the more two media, 1 and 2, are acoustically mismatched, the greater the reflection of ultrasonic waves at the media interface, and this effect is described by the reflection coefficient, $R_o = \rho_1 c_1 - \rho_2 c_2 / \rho_1 c_1 + \rho_2 c_2$ (1)

ACOUSTIC WAVE VELOCITY

In gases $c = \sqrt{\frac{\gamma P}{\rho}}$ --(2),

where γ is the ratio of principal specific heats, P the pressure, and ρ the density. As $\rho = m/V$, where m is mass and V is volume and PV = mRT, where R is a gas constant, it follows that c is proportional to $\sqrt{T}$, where T is the absolute temperature.

Where liquids are concerned, $c = \sqrt{\frac{K}{\rho}}$ --(3),

where K is the bulk modulus and the liquid compressibility is negligible. For liquids which are compressible, ethyl ether, for example, γ, the specific heat ratio is required and $c = \sqrt{\frac{\gamma K}{\rho}}$ --(4).

In solids, $c = \sqrt{\frac{Y}{\rho\ (1-b)}}$ --(5),

where Y is Young's Modulus and ρ is density and $b = 2\sigma^2/1-\sigma$, where σ is Poisson's Ratio.

ACOUSTIC WAVE ATTENUATION

Acoustic wave attenuation or losses in gases, liquids and solids are very complex and depend on molecular absorption, viscosity and heat conduction, and theory and experimental results do not agree well. However, Stokes[10] and Kirchhoff[11] have considered the absorption of sound through a medium due to heat conduction and internal friction and developed the following equation:

$$\text{Attenuation} \qquad 2\alpha = \frac{4\pi^2 f^2}{c^3 \rho_0}\left[\frac{4}{3}\eta + \frac{(\kappa - 1)}{c_p}k\right] \qquad (6)$$

where η is the coefficient of viscosity, f the frequency, ρ_o the mean density, c the velocity of sound in the medium, K the ratio of the specific heats, Cp the specific heat at constant pressure, and k the thermal conductivity. Apart from attenuation trends predicted by this equation, the attenuation of acoustic waves versus frequency is generally approximately proportional to f^2 for gases and liquids, and more directly proportional to f for solids.

Equation (6) does predict a direct effect of viscosity on the acoustic wave attenuation, an inverse effect of density (note ρc is acoustic impedance) and an inverse cubic effect of sound velocity.

APPENDIX C
Acoustic Waveguide Viscosity Measurements

A major advantage of embedded AWG is the ability to sense resin viscosity changes occurring over extremely wide ranges, for example from 10^2 Poise to 10^{11} Poise. Consequently, time was spent calibrating the AWG system so that meaningful viscosity measurements could be made. These measurements which are described next, were carried out using castor oil as the calibrating medium because of its large and known viscosity changes at low temperature.

During AWG cure monitoring both the signal attenuation (α in nepers, or the logarithm to the base e of the ratio of the initial signal to the signal at time (t)), and wave velocity (c in m/sec) are related to resin viscosity prior to gelation and to the resin modulus after gelation. In order to further understand this relationship and be able to approximately determine resin viscosity values from the AWG measurements some previous data from related AWG projects have been analyzed. These data of AWG attenuation were taken using a 10 cm length of 10 mil diameter Nichrome AWG embedded within castor oil taken to -49°C, Figure C1. In this Figure, the values of AWG attenuation in nepers/10 cm are plotted versus the logarithm of the absolute temperature. It can be seen by the changes in slope that phase changes occur within the castor oil as it freezes. Specifically, at 5°C, -26°C and -46°C. This is a phenomenon which also occurs within water as it freezes.

The most important part of the Figure C1 curve is the region from +5°C to -20°C where the attenuation data follows a straight line plot. In this region, the actual values of castor oil viscosity are known from the literature, and are plotted in Figure C2 in the form of logarithm of viscosity versus logarithm of the absolute temperature. The plot of these data approximates to a straight line as is the case with most fluids. Also, in Figure C2, the data for AWG attenuation (α) are plotted versus the logarithm of the absolute temperature. These attenuation data form an approximate straight line plot which parallels the viscosity plot. Obviously, there is a direct relationship between AWG attenuation (α) and the logarithm of viscosity (P), and this relationship is illustrated in Figure C3. It can be seen that the relationship is linear with a unity slope and a law, $\alpha = \log P - 1.323$, which can be transposed to viscosity (P) = $10^{\alpha+1.373}$ Poise. Consequently, for the special conditions of a 10 cm long, 10 mil diameter Nichrome AWG embedded within a curing resin or a changing viscous liquid, an approximate value of viscosity can be estimated from the attenuation value α. Based on the measured attenuation factor (α) from different materials the following estimates, Table C1, of the viscosity values at gelation (the transition from a liquid state to that of a rubbery gel) were made.

Table C-1 (also shown in Section 5.7 as Table 5-9)
Estimated Viscosity Values at Gelation:

MATERIAL:	VISCOSITY: (Poise)
Jello	134
Polyurethane	2×10^5
815 Resin	10^6 to 3×10^7
Concrete	3×10^9
Polymer Concrete	10^{11}

It is important to note in the analyses just described that the Figure C-2 curve of attenuation (α) versus the logarithm of absolute temperature which approximates to a straight line, is also found for curing resins, and consequently allows activation energies to be estimated from the chemical reactions taking place before and after gelation. In addition, the attenuation (α) versus time for curing resins yields straight line curves which demonstrate first order chemical reactions. This is evident from the equation of Arrhenius which may be expressed as the number of molecules undergoing chemical change per second = constant x $e^{-E/RT}$, where E is the activation energy, R the gas constant, and T is the absolute temperature. It should be noted that the attenuation (α), which for the AWG measurement is logarithm to the base e of a changing signal ratio, is a valuable measurement parameter, as exponential changes are very common in chemical and physical processes.

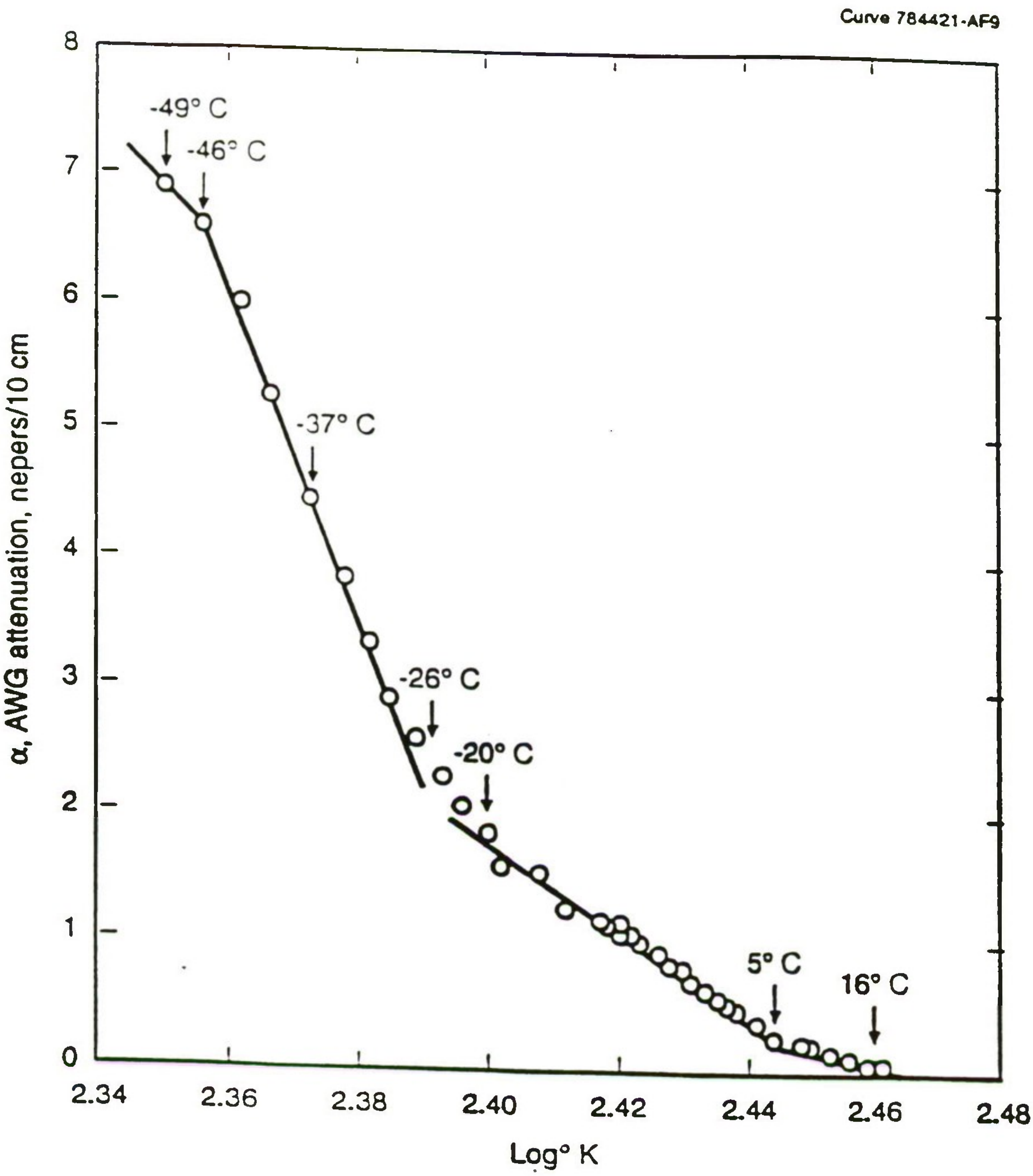

Figure C-1. Acoustic waveguide attenuation (nepers/10 cm) versus the logarithm of the absolute temperature for Castor oil taken to -49°C

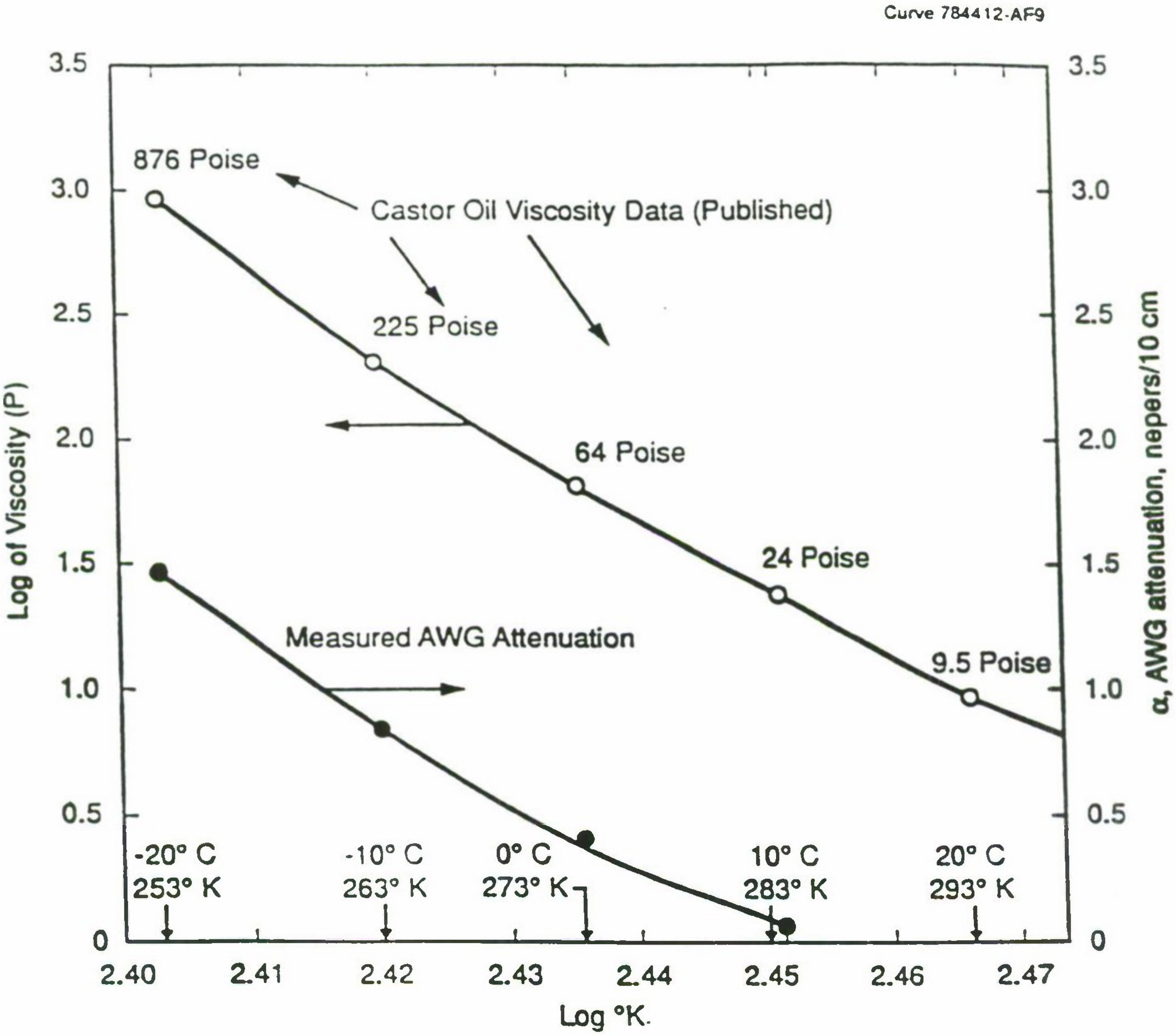

Figure C-2. Acoustic waveguide attenuation (nepers/10 cm) and logarithm of Castor Oil viscosity versus the logarithm of the absolute temperature

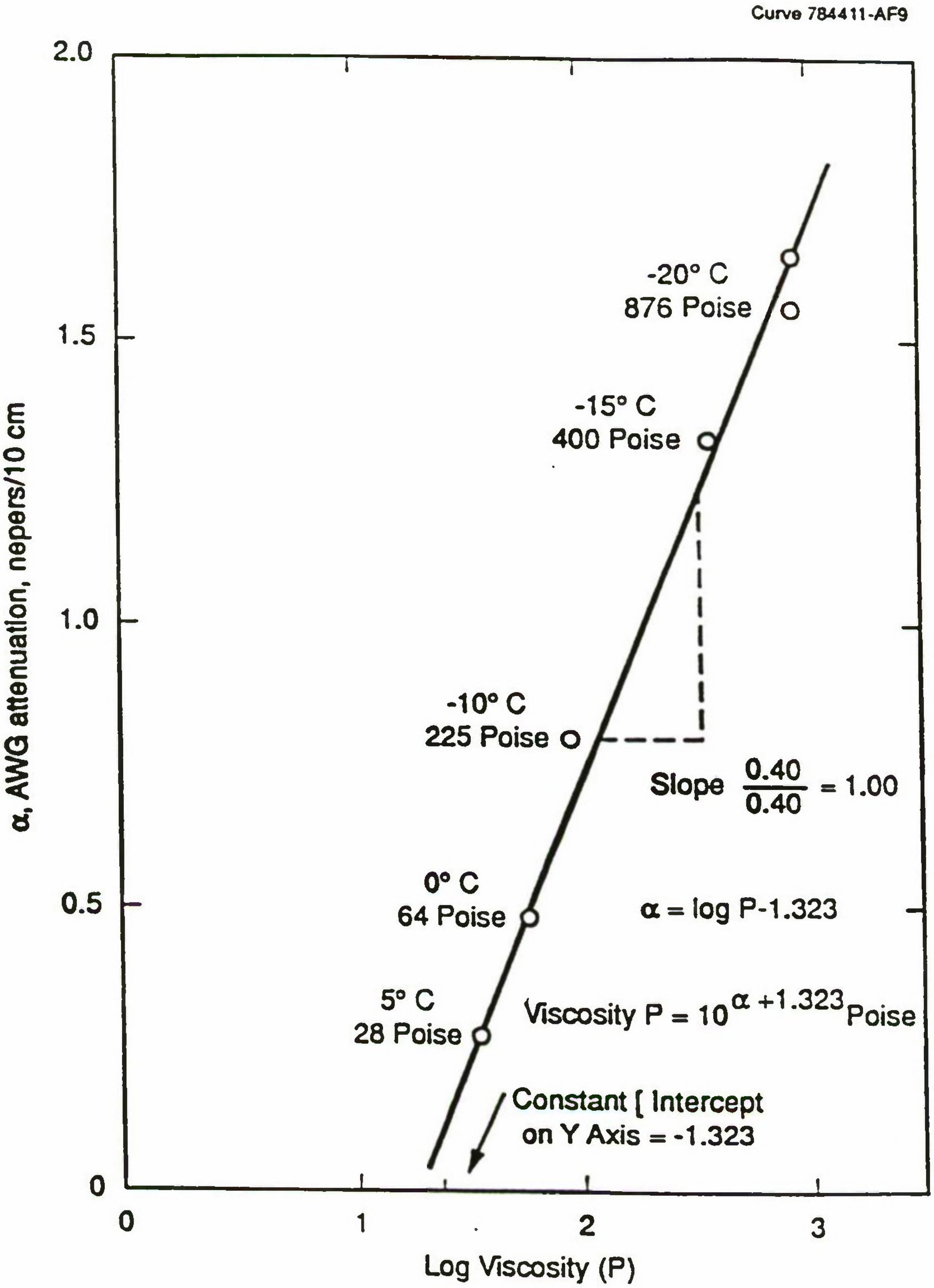

Figure C-3. Acoustic waveguide attenuation (nepers/10 cm) versus the logarithm of Castor Oil viscosity over the temperature range of 5° to -20°C

APPENDIX D
SCIENTIFIC PAPERS

During this two year acoustic waveguide development program, four scientific papers were co-authored by R. T. Harrold of Westinghouse Science & Technology Center and Major Richard Brynsvold of the U.S. Army, TACOM. These papers are listed below and copies of each are included within this Appendix:

1. "Embedded Acoustic Waveguides for Monitoring Composite Material Processing and N.D.E." presented at the Advanced Composites Conference, Dearborn, Michigan, 7-10 Nov 1994.

2. "Strain Measurements Inside Composite Materials Using Embedded Acoustic Waveguides", presented at the Conference on Nondestructive Evaluation Applied to the Process Control of Composite Fabrication, St. Louis, Missouri, Oct 4-5, 1994.

3. "Acoustic Waveguide Cure Curves for Materials Ranging from Fast Cure Resins to Slow Cure Concrete", presented at the 22nd Annual Conf. on the Review of Progress in Quantitative N.D.E., University of Washington, Seattle, Washington, July 30-August 1st, 1995.

4. "Acoustic Waveguide Sensing of Mold Filling and Bubble Formation During Liquid Composite Molding", presented at the Advanced Composites Conference, Dearborn, Michigan, Nov 6-9, 1995.

Embedded Acoustic Waveguides for Monitoring Composite Material Processing and NDE

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Reprinted from
ADVANCED COMPOSITES X
Proceedings of the 10th Annual ASM/ESD
Advanced Composites Conference
Dearborn, Michigan, USA
7–10 November 1994

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Library of Congress Cataloging Card Number: 94-78957
ISBN: 0-87170-542-7
SAN: 204-7586

ASM International®
Materials Park, OH 440730002

Printed in the United States of America

Proceedings of the 10th Annual ASM/ESD Advanced Composites Conference, Dearbom, Michigan, USA, 7-10 November 1994

Embedded Acoustic Waveguides for Monitoring Composite Material Processing and NDE

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Abstract

Embedded acoustic waveguides (AWG) of 10 mil diameter Nichrome for example, can be used to guide acoustic waves through composite parts during processing and the wave attenuation data can be interpreted in terms of real time viscosity, gelation and modulus information. Consequently, embedded AWG offer a means of feedback and control of composite molding processes. Following the manufacture of a part, measurements of acoustic wave velocity can yield approximate measurements of internal residual strain and material modulus and help in quality assessment. During the material lifetime the embedded AWG are available for internal strain sensing (via attenuation) and modulus measurement (via wave velocity) which should help in assessing damage and remaining life.

In a program funded by the National Automotive Center* it is planned to refine and further develop this AWG technology for production line use during the manufacture of both military and civilian composite vehicles. Specifically, this program addresses the control of resin transfer molding processes with emphasis on monitoring complete cure, void formation, complete mold filling, bondline integrity and N.D.E. In the work reported here it is shown that the embedded AWG can monitor the cure of parts with right-angled bends, yield credible values for resin viscosity and modulus, and have the potential to determine the relative shear strengths of quasi kissing-type of bonds between resin parts.

ACOUSTIC WAVEGUIDES (AWG) EMBEDDED WITHIN MATERIALS allow the monitoring of material properties during manufacturing processes and also throughout the material lifetime (1 to 4). In addition, they can be used for non-destructive evaluation (N.D.E.), for example, for measuring internal strain and sensing and locating damage. The AWG may be of many sizes and materials, provided the AWG and host material have similar thermal expansion characteristics, and the acoustic waves may have different values of frequency. In the work reported here, 10 mil diameter Nichrome AWG were embedded within resin specimens and the transmitted acoustic signals had frequencies ranging from ~60 to 80 kHz. Although AWG theory can be very complex, their function within materials can be understood by considering the acoustic impedance (density (ρ) x acoustic wave velocity (c)) of both the host material and AWG. If the acoustic impedance of the host material matches that of the AWG, then only a small signal will be transmitted within the AWG as most

*. *Contract: DAAE07-93-C-R121*
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of the signal is attenuated. However, if the acoustic impedance of the host material is different to that of the AWG, then due to reflections at the interface, a larger signal level will be transmitted within the AWG and there is less attenuation. Consequently, during the processing of a material, such as a resin, as this changes state from a liquid to a highly viscous solution; to gelation (transition from a liquid state to that of a rubbery gel); to a more rigid material; to a high modulus state; the attenuation of signals within the AWG tracks these different states.

Several studies (1 to 4) relating to the use of embedded acoustic waveguides for monitoring material processes and properties have been reported on previously and most of these are related to flat panels of graphite-epoxy composite materials and straight lengths of AWG sensors.

In the new work reported on here the AWG sensor was used to monitor the cure of a complex shaped resin part, i.e., a part with two right-angled bonds; a more detailed analysis was made of AWG measurements of the resin viscosity and modulus; and the AWG were used to interrogate bondlines between resin parts with different degrees of bonding, or quasi-kissing bonds. A kissing bond, for example between two clear resin parts, would have the appearance of complete bonding, but in reality, a considerable area at the bondline may not actually be bonded, and consequently, the bondline would have a poor mechanical shear strength.

AWG Cure Monitoring of Resin Parts with Two Right-Angled Bends:

During this study, in order to reduce time and cost, and also to obtain practical data from a large number of experiments, a simple mold was designed and constructed for making small-size (10 cm wide with a 1 cm cross section) resin specimens with two right-angled bends and a single embedded AWG, Figure 1.

In Figure 1 it can be seen that the AWG exits at each end of the mold and the AWG terminations are bonded to acoustic transducers, one acting as a transmitter and one as a receiver. At the transmitter end, pulsed ~70 kHz wavetrains from a signal generator with a repetition rate of ~100 pulses per second are used to activate the transmitter.

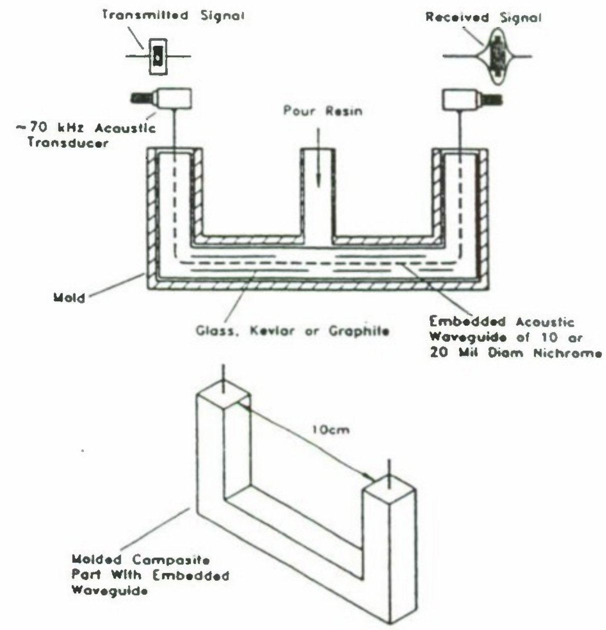


Figure 1. Arrangement for acoustic waveguide cure monitoring of molded composite part.

The acoustic transducer converts these electrical signals to acoustical pulses which are transmitted through the AWG to the receiving transducer at the opposite end of the AWG. Here, the acoustic signals are converted back to electrical signals and measured on an oscilloscope. The important measurement parameters are, the peak height of the received signal and the acoustic wave transit time from one end of the embedded AWG to the other. During resin processing, the changes in value of the received signal yield attenuation data which are used for monitoring the curing cycle. From the wave transit time data, the acoustic wave velocity within the embedded AWG can be obtained and these velocity data can also be used for monitoring cure. Usually, however, this is not as accurate a method as using the AWG signal attenuation, which is the preferred method for AWG cure monitoring.

Typical AWG cure curves of AWG signal levels and AWG wave velocity values for a Shell 815 resin (a thermosetting modified bisphenol-A epoxy resin) are shown in Figure 2. Note that instead of attenuation values, just the changes in the AWG transmitted signal levels with time are plotted on a logarithmic scale. Attenuation (α) at any time (t) into the curing process is readily obtained as it is simply the logarithm to the base e of the ratio of the signal at time (t) to the signal of time zero and is expressed in nepers. From the Figure 2 curves it can be seen that in the first part of the curing cycle, as the resin viscosity increases, the AWG transmitted signal level falls several orders of magnitude in value to reach a minimum at gelation (the transition from a liquid state to that of a rubbery gel). Following gelation, as the resin becomes more rigid and hardens the AWG transmitted signal level increases by two orders of magnitude.

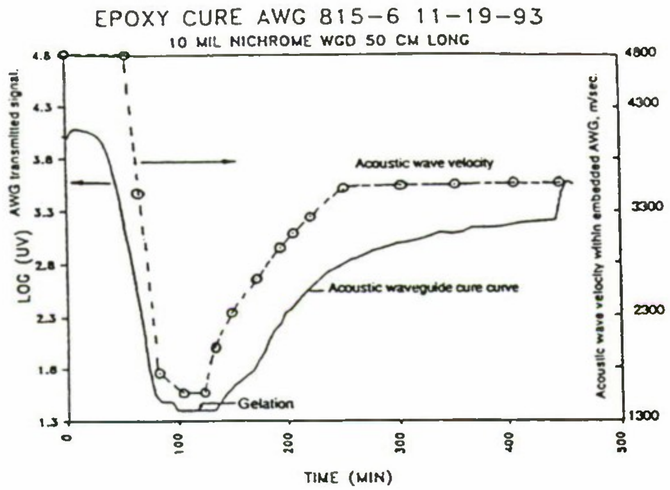


Figure 2. Acoustic waveguide cure curve for part with two right angle bends molded using Shell 815 resin with accelerator and heated via infra-red.

As explained earlier, the AWG transmitted signal changes during the cure cycle because the acoustic impedance of the resin is changing and restricting the transmission of signals within the acoustic waveguide. Actually, during the curing cycle the acoustic wave, which was initially travelling at a velocity of ~5000 m/sec within the AWG; travels at a slower velocity in the resin at the interface with the AWG surface, Figure 2. From Figure 2 it can be seen that during the cure cycle the initial acoustic wave velocity is ~4800 m/sec and this surprisingly falls to ~1300 m/sec at gelation, and near the end of cure at ~400 minutes, the velocity is near 3300 m/sec.

The important aspect of this study is that although the resin part has two right-angle bends which the embedded AWG must pass through, the sensitivity of the AWG is not diminished and the cure curve is similar to those found in previous studies[4]. Also, as the resin is clear and transparent, residual optical strain patterns can be viewed on a Polaroscope. In Figure 3, which is a picture of the residual strain patterns in the right-angle bends of resin specimens both with and without an embedded AWG, it does not appear that the AWG causes any strain problems. In addition, it has also been demonstrated that measurements of acoustic wave velocity also can be used to monitor the resin curing cycle. The experiments described and discussed were repeated using resin reinforced with ~27% by weight of "S" glass fibers, and a similar looking signal level cure curve obtained, although the acoustic wave velocity data were of different values. This would be expected due to a higher acoustic wave velocity in the composite material compared with resin only.

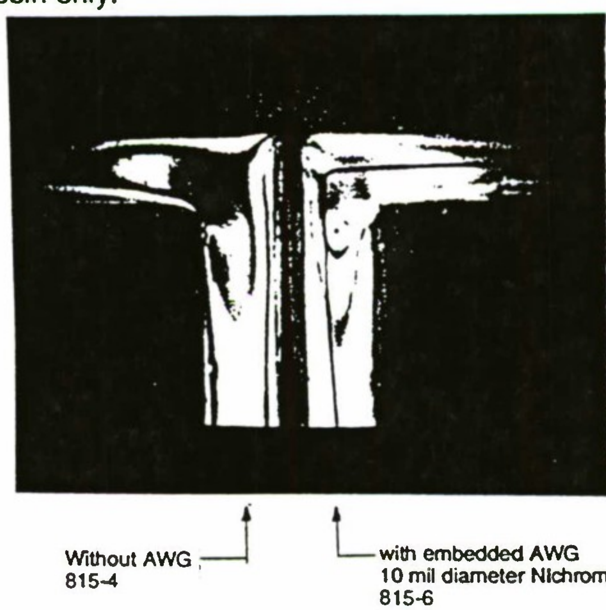


Figure 3. Residual strain patterns within molded right-angle specimens of clear Shell 815 resin both with and without embedded AWG.

Acoustic Waveguide Measurements of Resin Viscosity and Modulus

Resin Viscosity. It is a characteristic of curing resins that prior to gelation, they proceed through a period of rapidly increasing viscosity values extending over several orders of magnitude. As can be seen in Figure 2, the embedded AWG sensor readily provides a measure of these changes. However, in order to obtain calibration estimates for the actual resin viscosity values prior to gelation, a 10 cm length of 10 mil diameter Nichrome AWG was calibrated by measuring the reduction (attenuation in nepers) in value of the AWG transmitted acoustic signal as the castor oil was frozen to -49°C. Castor oil was chosen as its viscosity values, at least to -20°C, can be obtained from the literature.

The castor oil attenuation data were plotted against the logarithm of the absolute temperature, not shown here, and a linear relationship was evident, at least from +5°C to -20°C. This type of relationship is typical for most viscous fluids. Also, from prior studies[4] it is known that AWG signal attenuation ($\propto$) in nepers is related to resin viscosity prior to gelation and resin modulus after gelation. Consequently, for estimating resin viscosity values prior to gelation, it is reasonable to plot the castor oil values of AWG attenuation in nepers versus the logarithm of castor oil viscosity (P), and this resulted in the straight line quasi-calibration curve of Figure 4. From this curve the viscosity (P) equation $P = 10^{\propto+1.323}$ Poise, where $\propto$ is the AWG attenuation in nepers, can be derived.

Based on the measured attenuation values ($\propto$) from different materials and using the derived viscosity equation, the estimates, Table I, of the viscosity values at gelation (the transition from a liquid state to that of a rubbery gel) were made.

Resin Modulus. The AWG cure curves, Figure 2, illustrate the AWG response to increasing resin viscosity prior to gelation and to increasing resin modulus after gelation. A simple means of estimating the actual resin modulus values is to use the equation for the acoustic wave velocity;

$$c = \sqrt{Y/\rho} \ ,$$

Table I. Estimated Viscosity Values at Gelation

Material	Viscosity in Poise:
Jello	134
Polyurethane	2×10^5
Shell 815 Resin	10^6 to 3×10^7
Concrete	3×10^9
Polymer Concrete	10^{11}

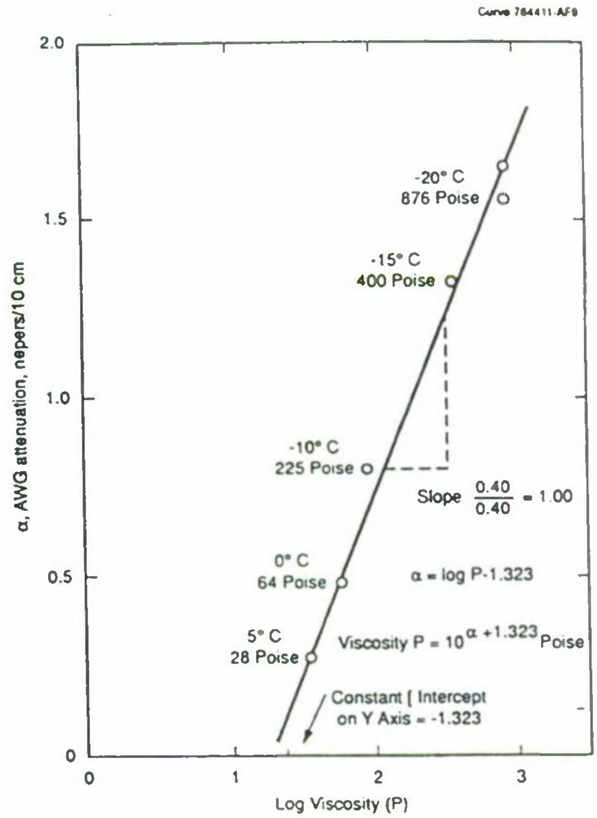


Figure 4. Acoustic waveguide attenuation (nepers/10 cm) versus the logarithm of Castor Oil viscosity over the temperature range of 5° to -20°C.

where c is the longitudinal wave velocity; ρ is density and Y is Young's Modulus. Actually, a term for Poisson's ratio is missing from the denominator of this equation, but that does not make a great deal of difference. The acoustic

wave velocity was measured within a resin part reinforced with 33% by weight of "S" glass fibers and was found to be ~5050 m/sec. Its density value was ~1.28 gm/cm^3 and from the velocity equation it was calculated that the reinforced resin part had a modulus value of ~32 GPa. Similar measurements on different materials yielded the modulus values, listed in Table II:

Table II. Estimated Modulus Values at the End of "Curing"

Material:	Modulus, GPa:
Shell 815 Resin	~ 8
Shell 815 Resin with 26.6% by weight of "S: glass fibers	~ 21
Shell 815 Resin with 33% by weight of "S" glass fibers	~ 32

AWG Sensing of Bondline Integrity:

A technical challenge in the science of materials is the bonding together of parts, especially where complex shapes and cylindrical structures are involved. One problem is that parts which have the appearance of being well bonded, or were processed to be well bonded, may have very little mechanical strength. These types of false bonds are called "kissing bonds". In applying the embedded AWG to the kissing bond problem it was thought that acoustic wave transmission between two AWG, one each side of a bondline could be used to assess the degree of bonding. Also, if the AWG were positioned parallel to, and close to (within one or two cm, or a wavelength) the bondline, then the signal transmission level along a single AWG may also respond to the bondline strength or degree of bonding.

In order to test this hypothesis, resin parts were molded with bondlines of varying bonded areas equispaced between two AWG embedded parallel to the bondline. This was achieved by first molding resin parts 1 x 2 x 10 cm with a 10 mil diameter Nichrome AWG passing through the center in the long direction, and then partially joining these parts with different areas coated with resin to form varying bondline adhesion areas, Figure 5. This configuration allowed acoustic interrogation of the bondline area, not only across the bondline from AWG to AWG, but also by transmission along each AWG located parallel to the bondline. Many interrogation paths are available, for example, using the AWG terminations marked 1, 2, 3, and 4 in Figure 5, acoustic transmission through the bondline can be from 1 to 4, 4 to 1, 3 to 2, 2 to 3, 2 to 4, 4 to 2 and from 1 to 3 and 3 to 1. Interrogation parallel to the bondline can be from 1 to 2, 2 to 1 and 3 to 4, 4 to 3. These types of measurements were made on twelve pairs of specimens bonded together with different degrees of bonding. The bonded areas ranged from zero bond (surfaces pressed together with an acoustic coupling agent, called Aquagel), to several different bonding areas with between 15% to 95% of the total area between parts bonded. The bonding was not precise as it was carried out by applying small drops of resin equispaced along the resin surfaces which were then pressed together and allowed to cure at room temperature. The areas covered by the resin spread as the parts were pressed together and after cure, as the resin is transparent the bonded areas were visually estimated. Next, acoustic interrogation measurements of the various bondlines of different bonding areas were carried out.

Two resin parts bonded together

Figure 5. Two resin parts, each with an embedded AWG, bonded together.

The maximum, average and minimum values of the acoustic data for transmission across the bondlines from AWG to AWG, are plotted against, the percentage of bonded area in Figure 6. Following the acoustic measurement, mechanical shear strength tests using the apparatus illustrated in Figure 7 were performed on all the bonded specimens and these data are also plotted in Figure 6. The most striking result which is apparent in Figure 6 is that resin parts with ~50% to ~70% of the area between them bonded have maximum shear strength, while parts with greater or lower percent bonding areas have less shear strength. Furthermore, most of the acoustic bondline measurement data correlates with the shear strength data.

Although these are encouraging results for demonstrating that AWG acoustic interrogation of bondlines can yield credible information in regard to the actual shear strengths of "kissing-type" of bonds, much more experimental confirmatory measurements are required. In addition, the fact that resin specimens with bondlines ~50% to ~70% bonded yielded maximum shear strengths, raises fundamental questions in regard to the bonding of composite materials. Preliminary analysis of the fractured specimens suggests tensile failure of the bonds and not shear failures. It is thought that this was due to the brittle or glassy nature of the resin used for bonding the parts together. Consequently, it is planned to repeat the tests using a less glassy bond in order to obtain true shear failures.

The data taken for signal transmission along AWG parallel to the bondline are not shown here, but were similar to those plotted in Figure 6 for transmission from AWG to AWG. These data are important because a single AWG embedded within a composite material would not only have the potential to monitor cure and lifetime NDE, but also be able to measure bondline integrity. For example, for inspection of a composite cylinder with a reinforcing ring bonded inside, an AWG could be embedded circumferentially either within the cylinder or the ring.

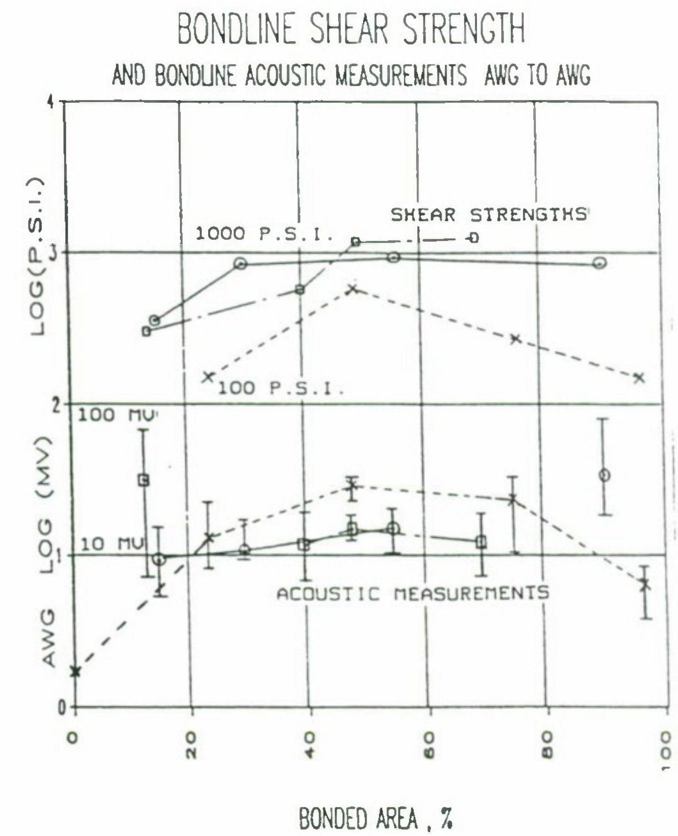
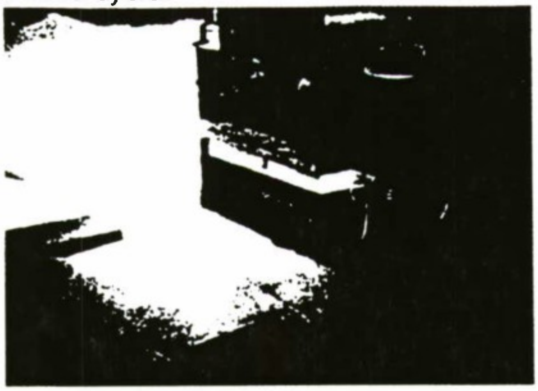


Figure 6. Comparison of bondline shear strength and measurements of acoustic wave transmission through bondline, AWG to AWG, versus percent bonded area for pairs of resin parts bonded together.

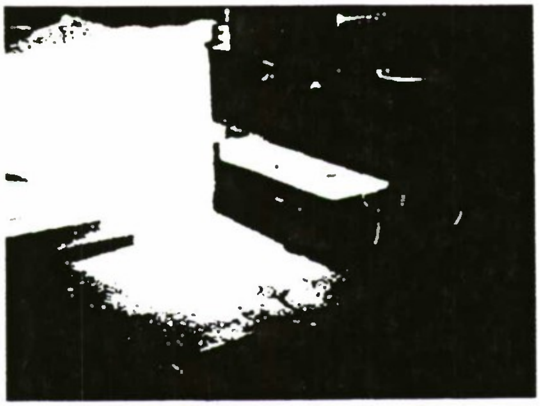
Conclusions and Discussion:

It has been demonstrated that the acoustic wave signal changes or attenuation within an embedded acoustic waveguide can be used for monitoring the cure cycle of resin parts with two right-angled bends. Even though the embedded AWG had to pass through the right-angles, its sensitivity was not reduced, and it responded to the orders of magnitude changes in resin viscosity prior to gelation and similar changes in resin modulus after gelation. In addition, it was

found that the values of acoustic wave velocity within the embedded AWG also gave a measure of the cure cycle.



Steel plate is placed on specimen surface prior to shear strength test.



Resin specimen clamped in position for testing.

Figure 7. Bonded resin specimen clamped in position for shear strength test.

Based on AWG measurements of castor oil of known viscosities and taken to -20°C, an equation was derived for estimating the viscosity values of resins and other materials at gelation. This yielded credible estimates for several materials. Modulus values of resins and other materials after gelation and at the end of cure were estimated using measured acoustic wave velocities and density values and the equation for longitudinal wave velocities in materials, and credible values were obtained for several materials.

The most important part of this study may be the AWG interrogation of bondline integrity in an attempt to measure and locate "kissing-type" or false bonds with little mechanical strength. Initial experiments for both bondline interrogation across a bondline from one embedded AWG to another, and for interrogation by examining signal transmission along an embedded AWG close to and parallel to a bondline, generally have a good correlation with the bondline mechanical shear strengths. The most striking result is that parts with approximately 50% to 70% of the area between them bonded have the highest shear strengths, while parts with greater or less percent bonding area have less strength. The correlation between acoustic measurements and bondline shear strength (or tensile strength) is encouraging, but much more experimental confirmation measurements are required before it can be claimed that AWG sensing systems can identify kissing bonds. In addition, as noted earlier, the tests need to be repeated with a less glassy bonding material in order to obtain true shear failure.

ACKNOWLEDGMENT:

Ed Diaz of (W) STC for mechanical test analysis and facilities.

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CONFERENCE ON NONDESTRUCTIVE EVALUATION APPLIED TO PROCESS CONTROL OF COMPOSITE FABRICATION:

St. Louis, Missouri, October 4-5, 1994

"STRAIN MEASUREMENTS INSIDE COMPOSITE MATERIALS USING EMBEDDED ACOUSTIC WAVEGUIDES"

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ABSTRACT

Acoustic waveguides (AWG) of 10 mil diameter Nichrome for example, can be embedded within composite materials during their manufacture for monitoring curing parameters and for providing sensing signals for process control. Following the manufacture of a composite part the embedded AWG can then be used during the material lifetime for measuring internal strain and for sensing and locating damage. In the work reported here which was performed under a contract with the U.S. Army Tank Automotive Command*, the embedded AWG were used to measure both the compressive and tensile strains within a stressed glass reinforced resin part.

It was found that as the part was subjected to increasing strain up to 2500 μs the attenuation of signals transmitted through the embedded AWG correlated with measurements from surface mounted strain gauges. In this paper, simultaneous measurements of AWG attenuation, strain gauge readings and optical strain patterns are reported on and discussed.

* Contract: DAAE07-93-C-R121
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1. INTRODUCTION

Acoustic waveguide (AWG) technology which has been developed over the last four decades covers a broad range of applications, such as; measurement of the elastic constants of materials;[1]; polymer cure monitoring[2]; radio frequency(R.F.) delay lines[3]; viscosity measurements[4]; measurements of the elastic constants of polymers undergoing solidification[5]; and measurement of a variety of physical phenomena[6]. More recently, AWG have been embedded within graphite-epoxy composite materials for both cure and structural health monitoring[7,8,9]. In an ongoing program for the U.S. Army (TACOM)*, the AWG are being applied to the control of resin transfer molding processes with emphasis on monitoring complete cure, void formation, complete mold filling, bondline integrity and N.D.E. The part of this study reported herein covers N.D.E. work, specifically, the application of embedded AWG for measuring strain within molded epoxy parts. It is shown that changes in value of signals transmitted within embedded AWG correlate with surface strain gauge measurements for molded epoxy resin parts subjected to both tension and compression.

2. MOLDED RESIN PART AND EMBEDDED ACOUSTIC WAVEGUIDE CURE MONITORING

In order to help in understanding the operation and measurement function of embedded AWG, before describing strain measurements, an outline is given of the type of resin parts used in this study and basic AWG cure monitoring. A simple mold, similar to that illustrated in Figure 1, was used to produce small resin specimens with two right-angled bends. The idea was to obtain practical data from a large number of easily produced parts. Right-angled bends were included to ensure that the embedded AWG would function within parts of complex shape. The embedded AWG was 10 mil diameter Nichrome, positioned within the mold as shown in Figure 1, with an ~70 kHz acoustic transmitter and receiver at opposite AWG terminations.

The AWG system is operated by using a signal generator to supply pulsed ~70kHz wavetrains at a rate of ~100 pulses per second to activate the acoustic transmitter. At the transmitter, the electrical signals are converted into acoustic pulses which travel through the AWG to the opposite termination where they are converted back to electrical signals and displayed oscillographically. The key measurement parameters are the peak height of the received signal and the acoustic wave transit time, from which the acoustic wave velocity can be obtained. These parameters are important for cure monitoring as they are related to the resin viscosity and modulus. Typical cure curves are shown in Figure 2.

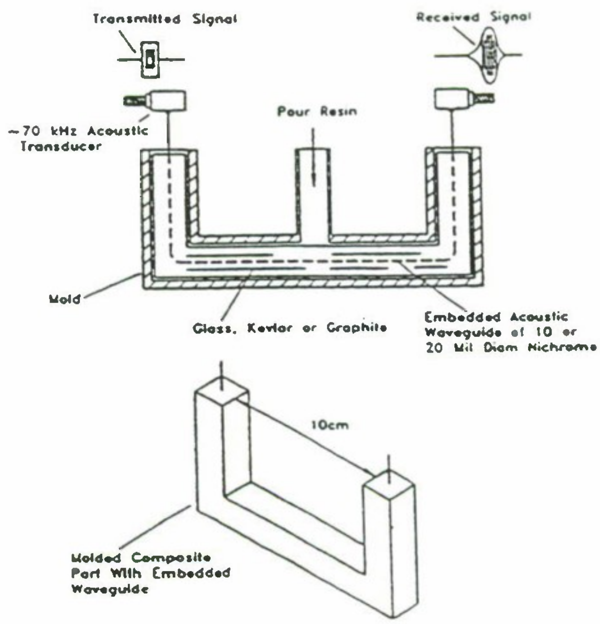


Figure 1. Arrangement for acoustic waveguide cure monitoring of molded composite part.

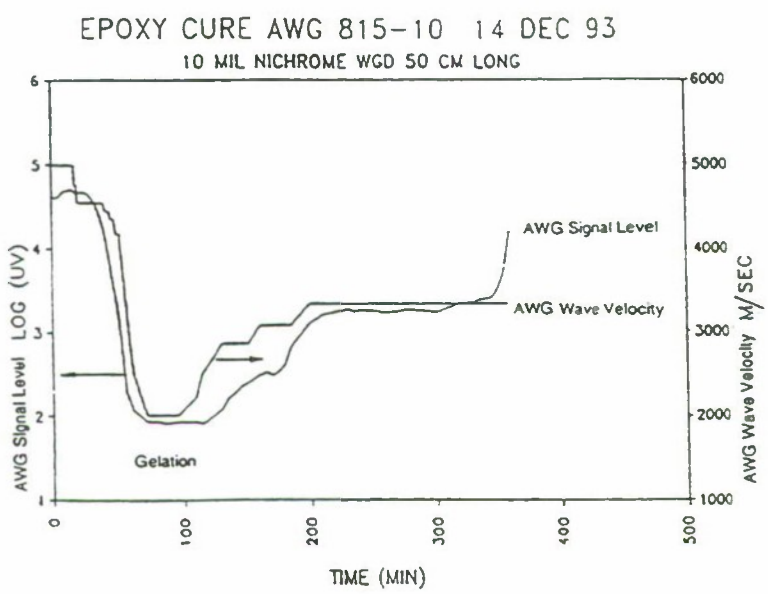


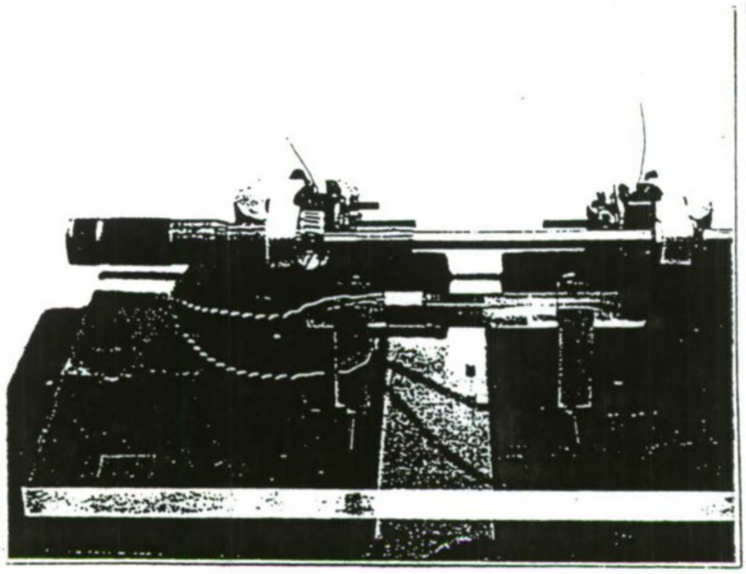
Figure 2. Acoustic waveguide cure curves (AWG signal level and velocity) for part with two right angle bends molded using Shell 815 resin with accelerator and heated via infra-red.

In order to understand the AWG function, it is helpful to consider the implications of the acoustic impedance of both the AWG and host material. Acoustic impedance (ρc) is simply the product of density (ρ) and acoustic wave velocity (c) for a given material. Consequently, if an AWG is embedded within a host material of similar acoustic impedance, very little signal will be transmitted through the AWG. On the other hand; if there is a large difference between the acoustic impedances of the AWG and host material, then, due to reflection at the interface, a large signal will be transmitted within the AWG as there is little signal loss at the interface. If the acoustic impedance of the host material changes during processing or during its lifetime, more or less signal will be transmitted within the AWG which is a major advantage of this type of sensor. During cure monitoring, Figure 2, the AWG falling signal levels are related to the resin viscosity values before gelation (the transition from a liquid state to that of a rubbery gel), and after gelation, the rising AWG signal levels are related to the resin modulus. In addition the acoustic wave velocity, Figure 2, which is actually the wave velocity at the interface between the AWG and resin, is related to the resin modulus.

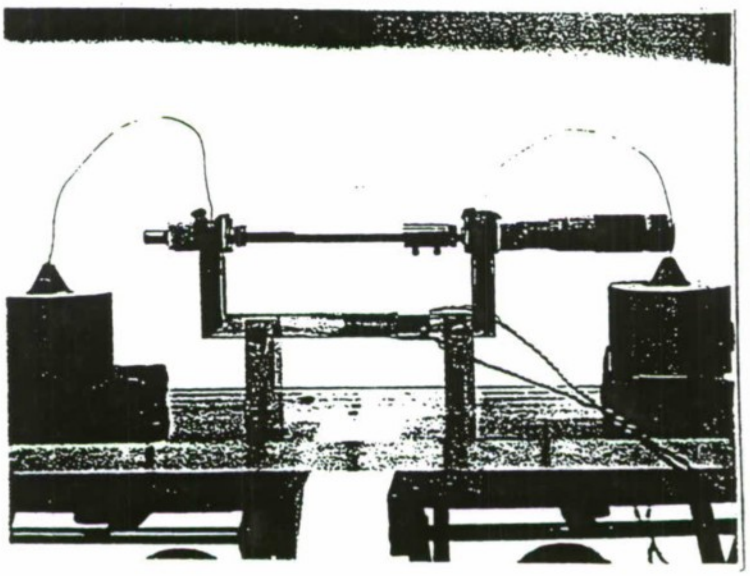
3. STRAIN MONITORING USING BOTH SURFACE MOUNTED STRAIN GAUGES AND EMBEDDED ACOUSTIC WAVEGUIDES

The clear resin specimens used for strain tests were 10 cm wide with a 1 cm x 1 cm cross section and two right-angled bends as illustrated in Figure 1. Conventional strain gauges were bonded to either the top or bottom surfaces of the horizontal part of the specimens. Three specimens were examined, each with an embedded AWG in a different location within the horizontal part. The locations chosen were ~1 mm from the top surface of the resin specimen, ~1 mm from the bottom surface, and in the middle or neutral zone (strain-free when the specimen is strained). In order to introduce strain within a specimen it was only necessary to slightly push the two vertical arms of a specimen toward one another or slightly pull them apart. Precise control of strain within a specimen was achieved using a clamping arrangement and micrometer, as shown in Figure 3. For example, about 120 mil of movement (i.e. pushing the specimen vertical arms toward each other, or movement in the opposite direction) would yield a surface strain reading of approximately 2000 compressive or tensile microstrain.

This was the procedure used to strain the three specimens, one with an embedded AWG near the top surface, one with the embedded AWG in the neutral zone, and one with the embedded AWG near the bottom surface. For each specimen, as it was strained to ~2000 microstrain in both tension and compression (as measured by surface mounted conventional strain gauges), measurements were made of the magnitude of the AWG transmitted signal, Figure 4. These curves show that for the AWG close to the top surface of a specimen, the AWG



VIEW SHOWING RESIN SPECIMEN
AND SURFACE STRAIN GAUGE WITH
MICROMETER FOR ADJUSTING STRAIN



VIEW SHOWING STRAINED RESIN
SPECIMEN AND AWG BONDED TO
ACOUSTIC TRANSMITTER AND RECEIVER

Figure 3. Pictures showing strained clear resin specimen with embedded AWG, surface strain gauges and acoustic transmitter and receiver.

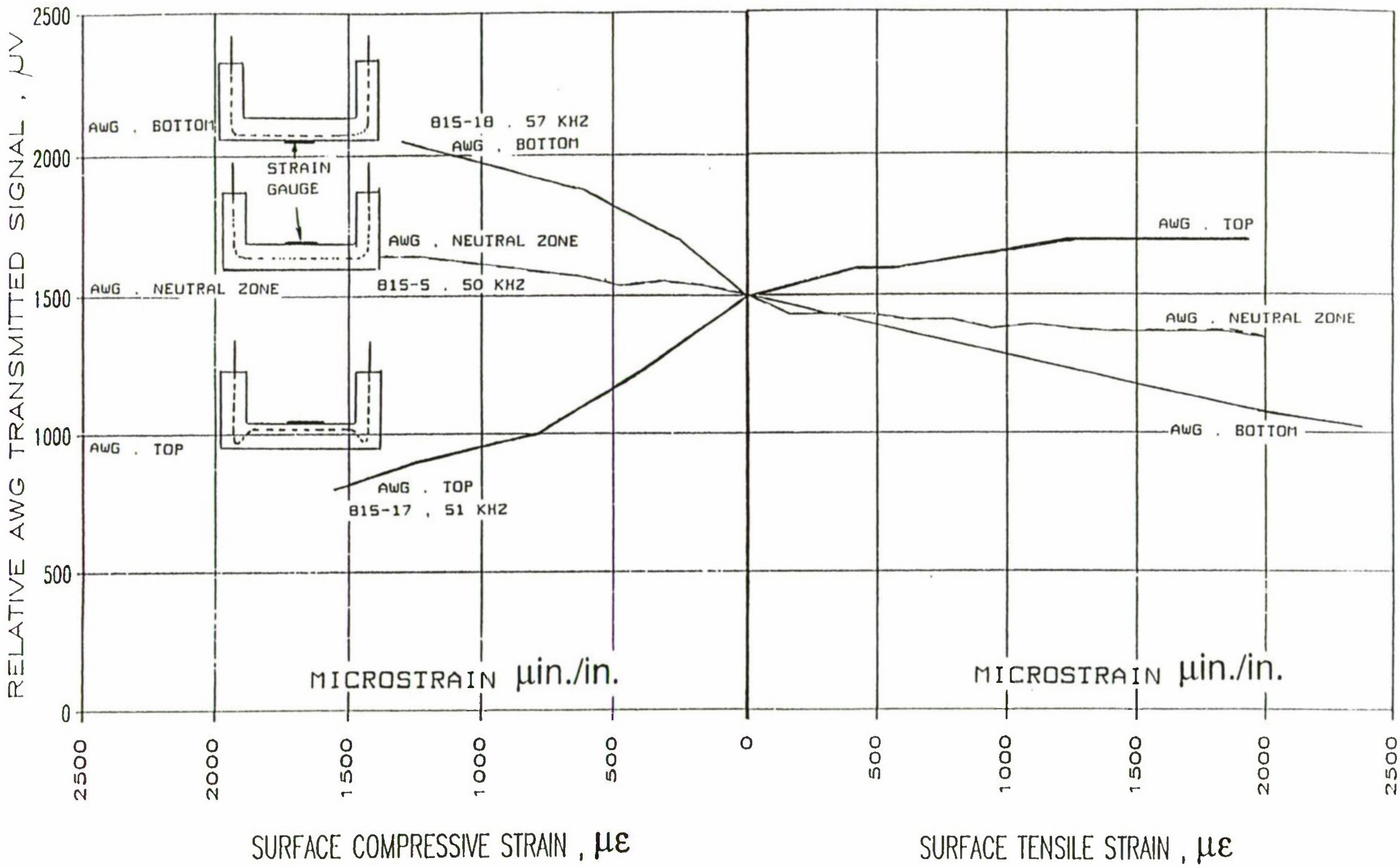


Figure 4. Comparison of relative embedded AWG transmitted signal levels and surface strain gauge readings for resin parts strained in both compression and tension.

transmitted signal falls under compression and increases under tension. While for the AWG embedded near the bottom surface, the AWG signal changes are reversed. With the AWG in the neutral or strain free zone, only small changes in the AWG transmitted signal occur under both compression and tension. It should be emphasized that these measurements indicate typical signal trends which are repeatable. However, actual values change on repeated testing, i.e., if the transmitted signal resonant frequency changes, or the AWG is bent at entry or exit which changes the terminal signal transfer function. The implication of these measurements will be discussed later.

In order to yield more information, optical strain patterns were also measured in a clear resin specimen strained to 2000 microstrain in both compression and tension, Figure 5. Although these TV screen pictures are not very clear they do show increasing numbers of strain fringes with increasing strain, and the compression strain patterns appear to be the reverse of the tensile strain patterns.

4. DISCUSSION

It has been demonstrated that changes in the transmitted signal levels within AWG embedded in resins strained in both compression and tension, show a correlation with surface mounted strain gauge readings to around 2000 microstrain. The AWG signal changes are not large, around 15 to 50% over the strain range, 0 to ~2000 microstrain, and changes in terminal transfer function will change actual readings; nevertheless, the signal trend with strain is repeatable.

The exact mechanisms which cause signal changes in an embedded AWG within a resin part subject to strain are not fully understood. It is known that for a new embedded AWG and specimens cycled several times through either compression or tension that the AWG transmitted signal level may reduce about 25% before it stabilizes. This is thought to be due to more intimate contact between the AWG surface and host material. Also, the level of the embedded AWG transmitted signal will change if the acoustic impedance (ρc) of the host material changes. It is not thought that the density (ρ) of a host material will change appreciably under strain, but the acoustic wave velocity (c) would change if the modulus of the host material changed under strain. Another area of concern, Figure 4, is that for the top AWG the AWG signal level fell under compression and increased under tension, while for the bottom AWG the signal changes reversed, rather than being the same as those for the top AWG. The AWG signal levels in the neutral zone, under both compression and tension are only small, as would be expected for an AWG situated in the neutral or strain free zone. The use of embedded AWG for measuring the internal strain within materials appears promising, but considerably more experimental work is required to theoretically understand the phenomena involved.

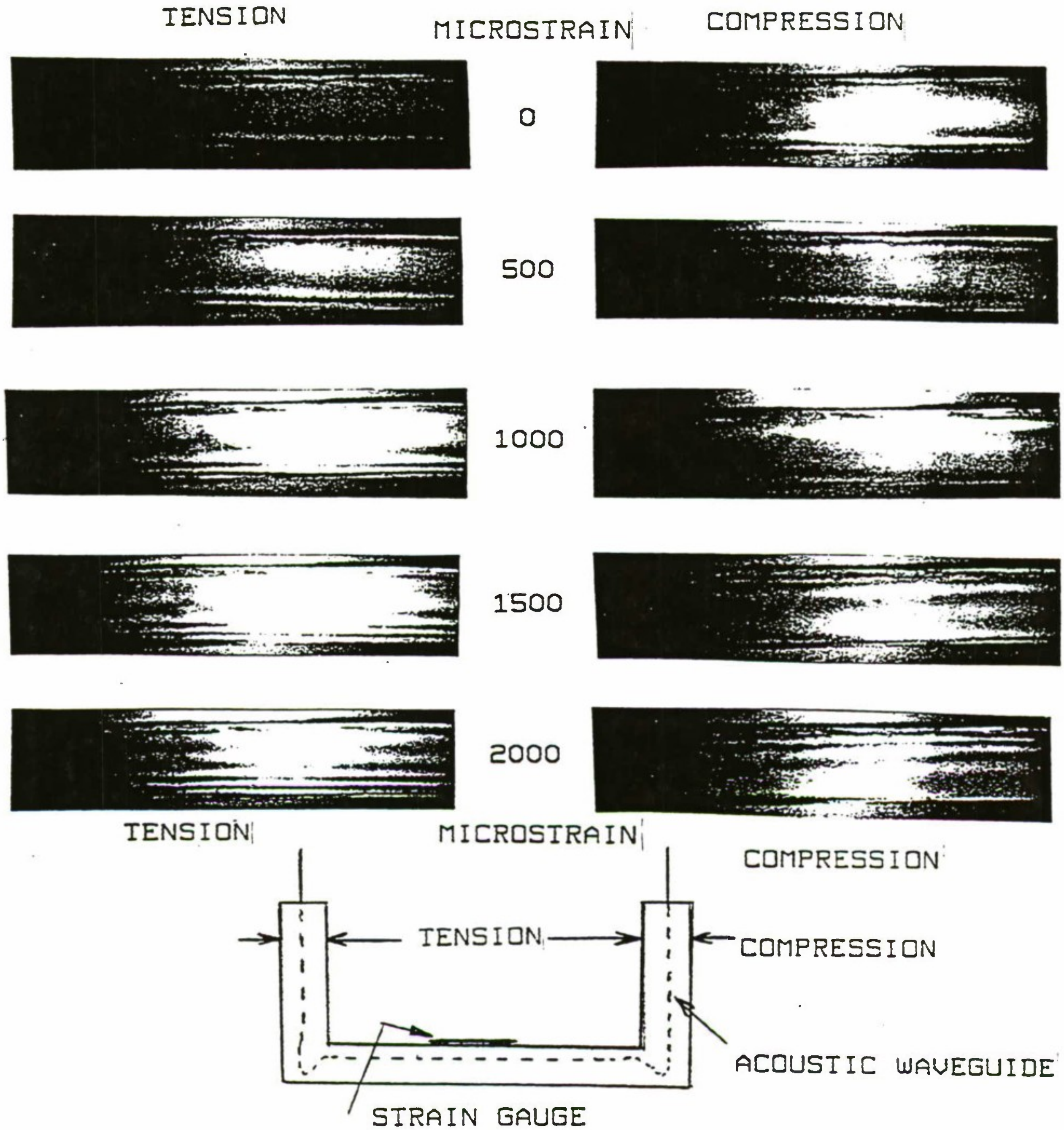


Figure 5. Optical strain patterns within clear resin specimen stressed to 2000 surface microstrain in both compression and tension.

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2/22/95

Abstract of paper for the Twenty-second Annual
Review of Progress in Quantitative Nondestructive Evaluation
Seattle, Washington, July 30th - August 4th, 1995

"ACOUSTIC WAVEGUIDE CURE CURVES FOR MATERIALS RANGING FROM FAST CURE RESINS TO SLOW CURE CONCRETE"

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ABSTRACT:

Over the last decade the technology of embedded acoustic waveguides (AWG) has evolved and been applied to cure monitoring of a variety of materials ranging from fast (minutes) curing resins to very slow (~ month) curing concrete. Although all the theoretical aspects of embedded AWG are not fully understood, sufficient data have been obtained to clearly show the three general phases of curing for all these materials, i.e., viscosity, gelation, modulus.

In this paper, data from embedded AWG cure monitoring measurements covering a wide variety of materials are presented and the common AWG features of attenuation and velocity changes and their relationship to viscosity, gelation and modulus are discussed. In addition, similarities between embedded AWG monitoring of curing materials and solidifying polyethylene, ice, and metals are outlined.

Part of the work reported
was performed under
Contract: DAAE07-93-C-R121
National Automotive Center/U.S. Army
(TACOM)/Westinghouse STC
C.O.T.R.: Major Richard Brynsvold

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INTRODUCTION

The attenuation and transmission velocities of ultrasonic waves traveling through curing polymers are related to the three general phases of curing which can be categorized as; increasing viscosity; gelatinization (the transition from a liquid state to that of a rubbery gel), and hardening (increasing modulus). This should be expected because the acoustic wave transmission in materials depends upon their molecular structure which is related to the material viscosity, density and modulus. In 1952 Sofer and Hauser [1] passed 2.3 Mhz longitudinal ultrasonic waves through curing polymers and related attenuation and velocity measurements to the degree of cure. However, wave dispersion can be a problem with this technique and an improved approach is to use embedded acoustic waveguides to guide the ultrasonic waves through curing polymers. A step in this direction was taken by Roth and Rich [2] in 1953 who developed a 28 kHz ultrasonic technique for measuring viscosity during the polymerization of plastics. They used a thin metallic strip into which shear waves were magnetostrictively induced and when this strip was immersed in a viscous liquid, the attenuation of the shear waves traveling along the strip could be related to the liquid viscosity. This metallic strip can be considered to be an acoustic waveguide (AWG) excited and measured from one termination. Later, in 1971, Lynnworth[3] and in 1974, Papadakis [4] developed AWG techniques using magnetostrictive activation of torsional waves and applied their methods to curing polymers. In 1986, two of the present authors, Harrold and Sanjana [5] reported 60-80 kHz ultrasonic AWG cure curves for graphite-epoxy composite materials. This work involved transmitting longitudinal waves through AWG, for example, 20 mil diameter Nichrome, embedded within the composite material. The waves were transmitted from one end of the AWG and received at the other end, and wave attenuation and transit time (velocity) were measured. The attenuation data versus time yielded a cure curve which showed orders of magnitude increase in wave attenuation during increasing material viscosity, maximum attenuation at gelatinization, and one or two orders of reduction in attenuation during the hardening phase. This embedded AWG approach not only provides a sensitive cure

curve during material manufacture, but the embedded AWG can also be used for quality assessment after manufacture and lifetime N.D.E.

Following the application of embedded AWG to thermosetting composite materials in 1986, this technique has been applied to composite thermoplastics, and a wide variety of materials ranging from ice, Wood's metal, Jeweller's wax, silicone rubber and polyethylene, to fast (minutes) cure epoxi-patch, to long [>2 months] cure concrete. It is the purpose of this paper to compare the embedded AWG curing data obtained from this wide variety of materials in order to further understand the common phases of increasing viscosity, gelatinization, and hardening or increasing modulus. In addition, by studying the results for materials such as Jeweller's wax, Wood's metal and polyethylene which were melted and poured around the AWG and allowed to cool and solidify; and for water and castor oil frozen around the AWG and allowed to warm up; phase changes within the materials can be identified.

In this paper AWG "cure" curves for all the materials examined are displayed graphically for comparison and their common features and implications of the measurements are discussed.

AWG CURE MONITORING CONCEPT

The basic concept [6] of AWG cure monitoring is that the amount of sound transmitted through an embedded AWG is closely related to the difference between the acoustic impedance of the AWG and the acoustic impedance of, and the pressure exerted by, the host material which surrounds the AWG. The acoustic impedance of the medium which surrounds the AWG depends upon the density of the host material times the velocity of sound through it. Thus, any physical or chemical changes which affect the density of the host material or velocity of sound through it will alter the acoustic impedance and change the amount of sound transmitted through the AWG. Since the density and velocity of sound through a host material are affected by temperature, stress, strain and impact on the host material, all these things can be monitored by means of the AWG. It is even possible to obtain an approximate value for Young's modulus from the information obtained through the AWG. The transit time of the sound wave through the AWG is also affected by these factors and can also be used to monitor changes that occur in the host material.

THEORETICAL ASPECTS

The actual wave propagation modes and velocities along acoustic waveguides can be very complex and were first analyzed by L. Pochhammer [7]in 1876. Since that time a very large number of papers have been written on the subject. Papers by Redwood [8], 1959, and Thurston [9], 1978, help in the understanding of guided wave propagation. More recently, studies by Nicholson [10], 1991, and Nayfeh and Nagy [11], 1995, yield further insight into wave propagation in AWG both in air and within a host material.

In this present AWG work on cure monitoring it has been helpful to identify guided wave propagation by tuning the system so that the received acoustic pulse

duration time is at least 10 times longer than the transmitted pulse duration time, a condition first recognized by May [12] in 1960. In addition, during AWG cure monitoring the signal changes measured during the viscosity (η) and hardening (modulus (Y)) phases are generally related to the square root expressions below which are a simplification of equations developed by Roth and Rich[2] during their work with an ultrasonic AWG probe for viscosity measurements.

Viscosity - attenuation. proport. to $[\rho\ \eta]^{\frac{1}{2}}$. Modulus - attenuation prop. to $[\rho Y]^{\frac{1}{2}}$.

PRACTICAL APPARATUS AND TECHNIQUE

The general method employed for AWG cure monitoring of materials and composites is outlined in the schematic of Figure 1. It can be seen that the wave transit time (velocity) and the peak magnitude of the received signal are measured oscillographically and a video record of this display is also made for later analysis of any rapidly changing signals.

ACOUSTIC WAVEGUIDE "CURE" CURVES FOR MATERIALS RANGING FROM FAST CURE RESINS TO SLOW CURE CONCRETE

Over a ten year period embedded AWG cure curves have been obtained for a variety of resins, composites, plastics, metals and other materials. These AWG "cure" data are listed in Table I, the individual "cure" curves are shown in Figure 2, and for comparison purposes, all the curves are plotted in Figure 3.

DISCUSSION OF THE PHENOMENA SENSED DURING EMBEDDED AWG MONITORING OF CURING RESINS AND MELTING AND SOLIDIFYING MATERIALS

A common feature of the AWG cure curves, Figure 3, is the several orders of magnitude increase in wave attenuation during increasing material viscosity, maximum attenuation at gelatinization, and one or two orders of magnitude reduction in attenuation during the hardening phase. Study of the AWG cure

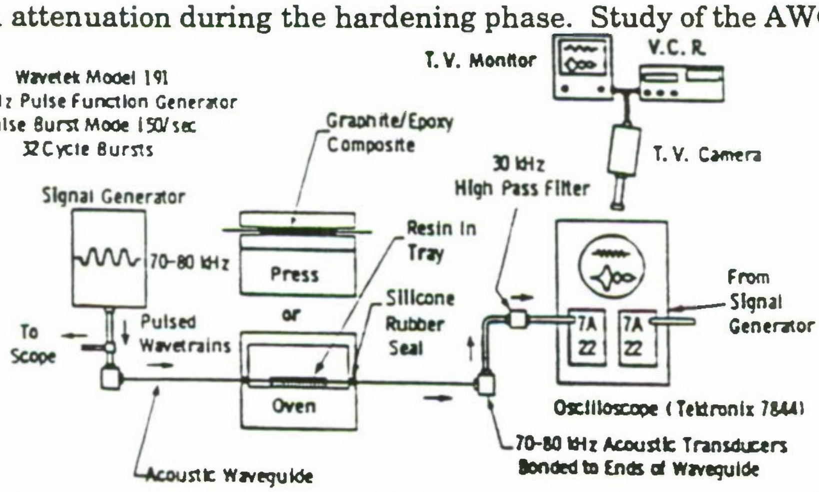

Figure 1. Schematic of apparatus for AWG cure monitoring of materials and composites.

Table 1
AWG "Cure" Data for Materials Ranging from Fast Curing Epoxy to Slow Curing Concrete

Material	Nichrome AWG	Embedded AWG	f kHz	Initial Wave Velocity, Poured m/sec	Wave Velocity Gelitinization m/sec	Final Velocity Hardening m/sec	Wave Velocity Material m/sec	Material Density gm/cm³	Modulus Est. from Velocity, GPa	Modulus From Lit. and Tables GPa
Epoxi-Patch Clear	20 mil x 10 cm	10 cm	70	5000	2500	2500	2000	1.18	~4.7	2.0
Shell Resin 815	10 mil x 50 cm	20 cm	67	5000	2000	3300	2200 (1660 liquid)	1.14	5.5	2.8
Graphite-Epoxy Thermoset 0/90-Hercules 3501-6AS	10 mil x 55 cm	32 cm	~70	4600	~3000	4600	6900	1.6	~76	~130 unidirectional
Graphite-Epoxy Thermoset 0/90 I.C.I.-T300/976	20 mil x 65 cm	20 cm	66	6700	~3300	3300	--	--	--	~130 unidirectional
Graphite-Epoxy Thermoplastic 0/90 APC-2 (PEEK)	20 mil x 100 cm	15 cm	71	3750	~650	3750	--	1.48	--	~130 unidirectional
Jello	10 mil x 30 cm	13 cm	68.5	4300	4300	4300	1370 + 12°C	1.06	1.9	$3x10^{7}$ to 1.5×10^{4} 1 to 20% gelatine
Water & Ice	10 mil x 30 cm	13 cm	60	4000 water	-1°C, 2600 -4°C to +4°C, 3000		3400, -36°C	0.92	8 to 11	9.0 -15°C to -50°C
Liquid & Frozen Castor Oil	10 mil x 10 cm	10 cm	64	3850, 24°C	2500, -49°C	3850, 24°C		0.95		--
Silicone Rubber R.T.V.	10 mil x 30 cm	13 cm	80	4000	4000	4000	800	1.14	0.73	1.5×10^{4} to 5.0×10^{4}
Syntactic Polyurethane	20 mil x 40 cm	20 cm	83	5000 Air 4160 Pour	4160	4160	860	0.58	0.43	0.01 to 0.02
High Density Polyethylene	20 mil x 30 cm	~1 cm	73	4200	4200 only one cm of AWG covered	4200	2300	0.94 to 0.98	5.0	0.3 to 1.0
Jeweler's Wax	10 mil x 20 cm	13 cm	64	5000	950	1500	2100	1.4	6.0	
Polymer Concrete (Polysil)	20 mil x 50 cm	12.5 cm	64	4800	2300	3400	4100	2.16	50	40
Polymer Concrete (with expanding monomer)	20 mil x 50 cm	12.5 cm	75	5000	1900	2550	3300	2.16	24	40
Concrete	20 mil x 56 cm	19.5 cm	68	5200	--	2240	~1000	~2.7-3.0	~3	20-35
Wood's Metal 50% Bi, 25% Pb, 12.5% Sn., 12.5% Cd by vol.	20 mil x 30 cm	13 cm	60	4400	500	1170	2060 to 2100	~10.0	44	10
Tin	10 mil x 30 cm	13 cm	67.5	4170	1030	1430	3300	7.3	~80	54

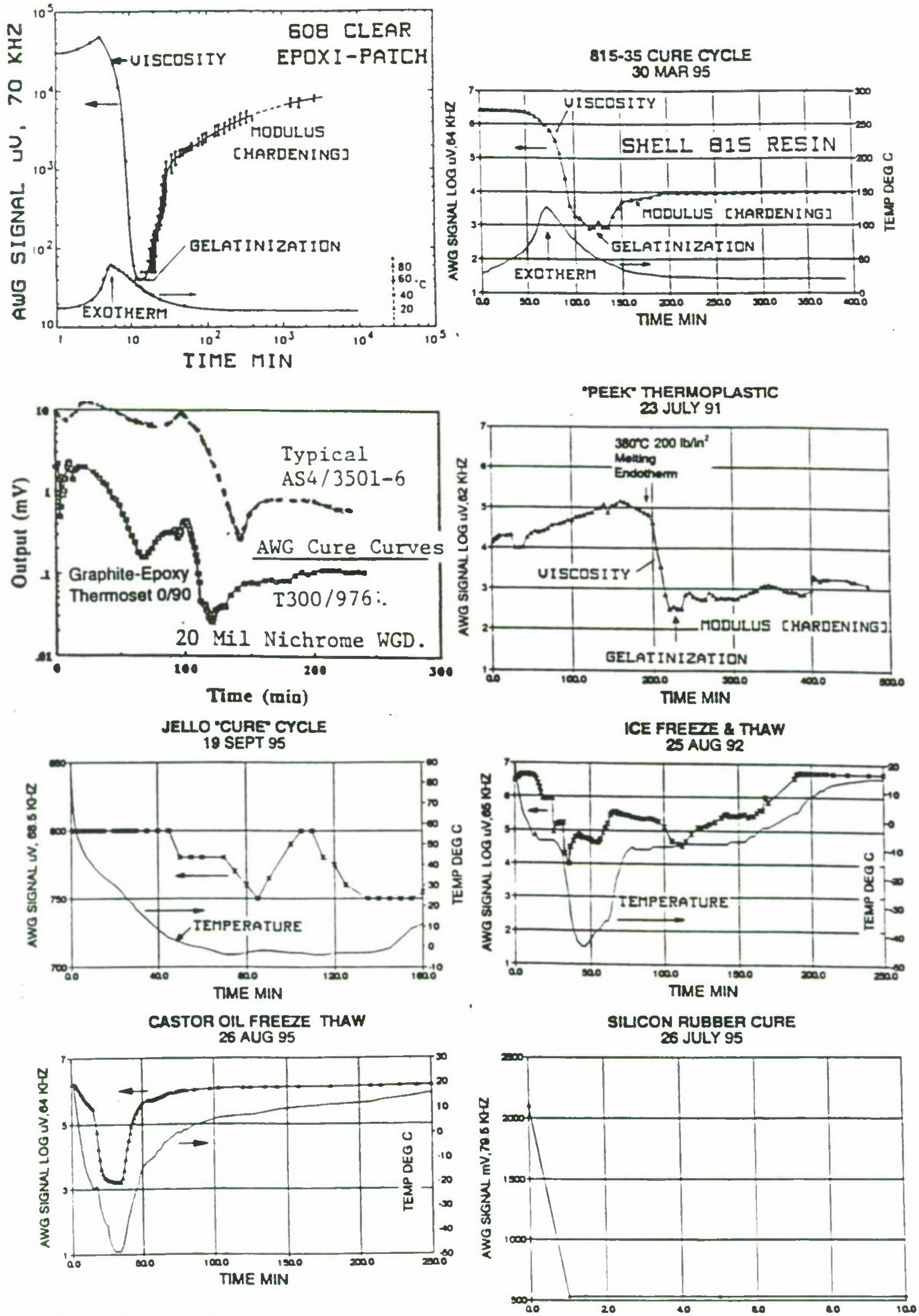

Figure 2. AWG "cure" curves.

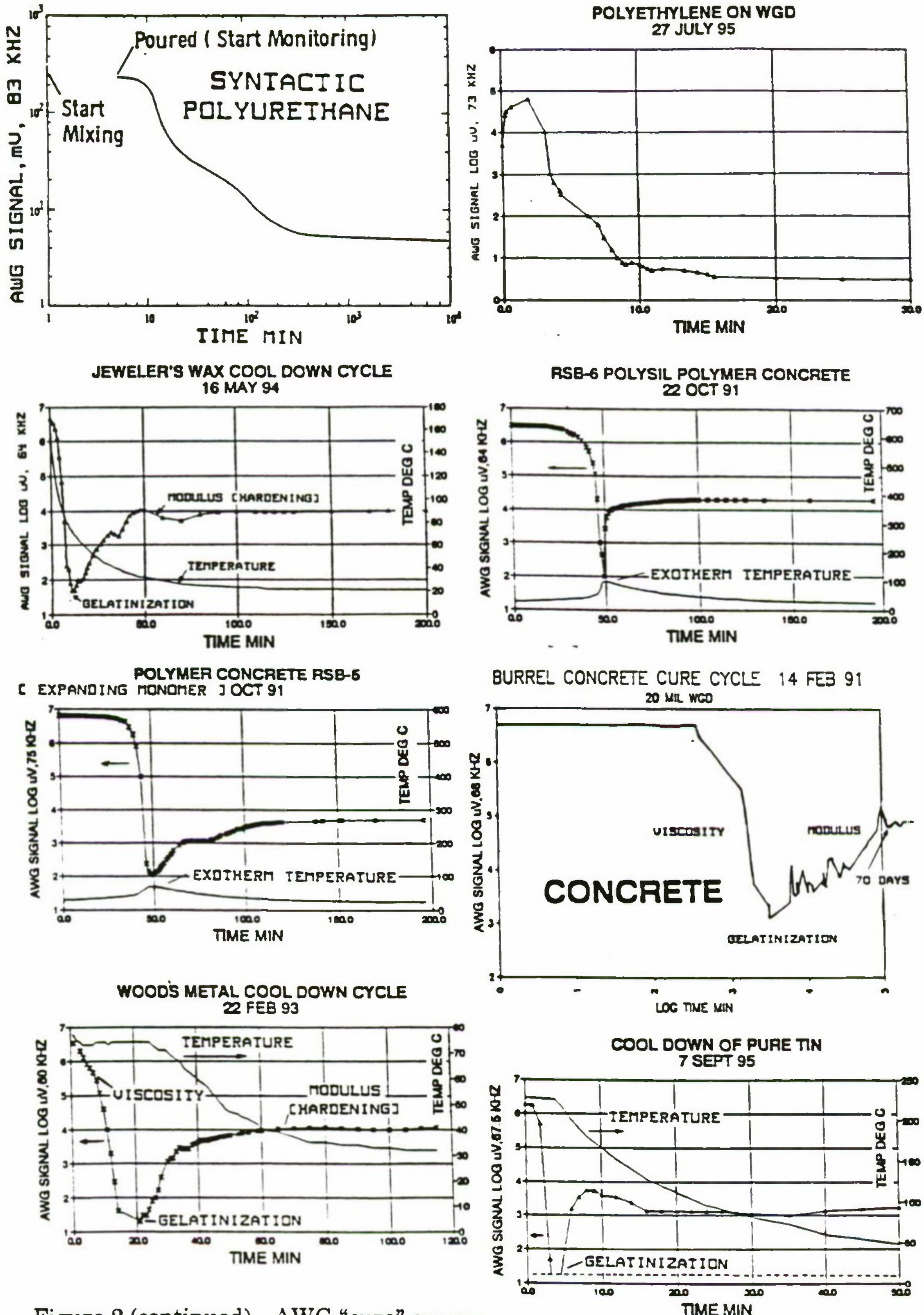

Figure 2 (continued). AWG "cure" curves.

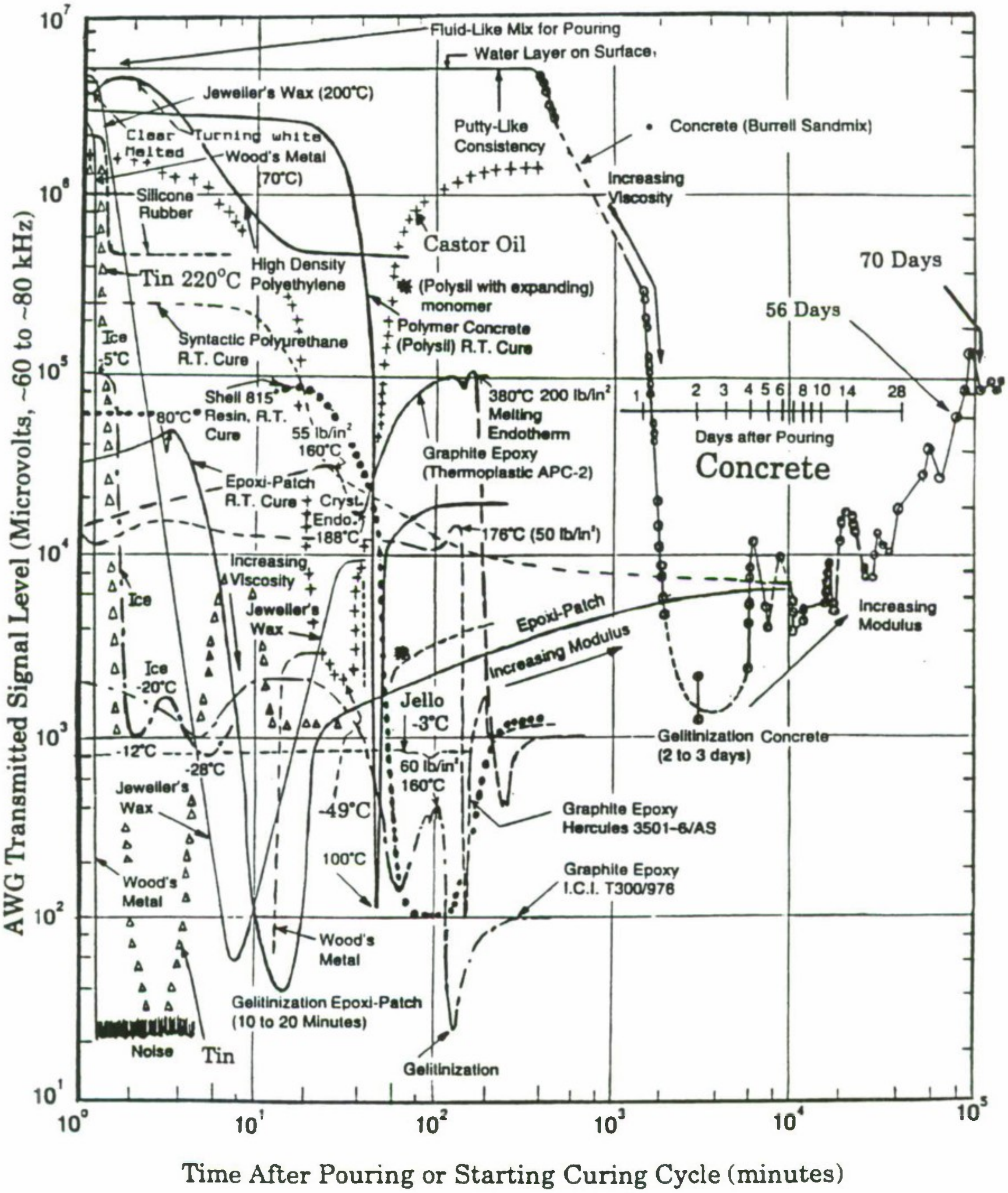

Figure 3. Embedded nichrome AWG "cure" curves illustrating the changes in the AWG transmitted signal versus time for various materials including fast curing Epoxi-Patch and slow curing concrete.

curves, Figures 2 and 3 reveals important features associated with several different materials as outlined below:

- With Epoxi-patch and Shell 815 resin, the peak exothermic temperature occurs at the start of the increasing viscosity phase and before gelatinization.
- In contrast, for polymer concrete, the maximum exothermic temperature coincides with gelatinization.
- It takes 2 to 3 days for gelatinization to occur within concrete and about 70 days to cure. Polymer concrete appears to cure in 200 minutes or less.
- Ice taken to -49°C exhibits phase changes (probably pressure changes) which can be identified by sudden changes in the "cure" curve at -12°C and -28°C, and also, hysteresis is evident. On the other hand, castor oil taken to -49°C exhibits a smooth "cure" curve.
- Polymer concrete with an expanding monomer should exhibit less shrinkage during cure and this is confirmed by the cure curves for polymer concrete with and without an expanding monomer. The polymer concrete (expanding monomer) cure curve indicates a lower AWG transmitted signal due to a better bonding (more pressure on AWG due to polymer expansion rather than shrinkage).
- With both Wood's metal and tin melted around the AWG, about six orders of magnitude of AWG signal reduction occur during the viscosity phase.
- Silicone rubber, polyethylene (small specimen, 1 cm around AWG) and syntactic polyurethane all act as signal damping materials and the usual AWG signal increase during material hardening is not evident.

ACKNOWLEDGMENT

Part of this work was performed under Contract DAAE07-93-C-R121. National Automotive Center/U.S. Army (TACOM)/Westinghouse STC. C.O.T.R. Major Richard Brynsvold/Dr. Basavaraju B. Raju.

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Abstract of Paper for the ASM/ESD 11th Annual Advanced Composites Conference & Exposition, Dearborn, Michigan, 6-9 November 1995

"ACOUSTIC WAVEGUIDE SENSING OF MOLD FILLING AND BUBBLE FORMATION DURING LIQUID COMPOSITE MOLDING"

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ABSTRACT:

Although the development of embedded acoustic waveguides is primarily being pursued for cure monitoring[1] of composite materials and lifetime N.D.E., these sensors can also be helpful for monitoring mold filling and the formation of gas bubbles. In work using a small transparent mold it was found that the Shell 815 resin flowed readily through the mold and around the glass fibers apparently without the formation of gas bubbles or voids. However, when the resin flow was speeded up using a partial vacuum, gas bubbles trapped beneath the lower surface of the mold were observed for several seconds flowing to the resin surface. These bubbles formed silently as no acoustic emissions in the ~3kHz to ~80kHz frequency range were detected. However, it is hypothesized that during these mold filling experiments non-visible gas bubbles formed during the exothermic temperature rise of the resin can be identified by an increase in the value of the AWG transmitted signal. Based on these experimental results, it is believed that embedded AWG will be helpful for determining when liquid molds are filled with resin and yield some information on whether gas bubbles are generated during mold filling. It is anticipated that for applications involving full size liquid molds several embedded AWG will be required.

[1] Contract: DAAE07-93-C-R121
National Automotive Center/U.S. Army (TACOM)/Westinghouse STC
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ACOUSTIC WAVEGUIDE SENSING OF MOLD FILLING AND BUBBLE FORMATION DURING LIQUID COMPOSITE MOLDING

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ABSTRACT:

In order to assess the performance of embedded acoustic waveguides[1] (AWG) for monitoring the quantity of resin within a liquid composite mold, and also the formation or presence of gas bubbles, some laboratory experiments were carried out. A small transparent mold was gradually filled with resin and the changes in level of the AWG transmitted signal did give an indication of the degree of mold filling. This system, although useful, was not sensitive enough to detect the presence of trapped gas which was revealed when a vacuum was applied. These gas bubbles apparently formed silently as no acoustic emissions (~3 kHz to ~80 kHz) were detected.

Additional experiments, directed toward understanding the AWG response to gas in the resin (without hardener) demonstrated that when the resin is presumably saturated with nitrogen gas introduced via an immersed perforated pipe, and no bubbles are visible within the resin, the acoustic signal level transmitted between immersed AWG is reduced by an order of magnitude. The original resin condition returns in about three hours. Test results for ginger ale (presumably saturated with carbon dioxide) were similar, but reversed, in that the initial AWG signal level fell an order of magnitude in value, from the maximum fizz to flat condition, over a two hour time period. This may be a function of the special ~60 kHz acoustic characteristics of carbon dioxide.

[1]Contract: DAE07-93-C-R121
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ACOUSTIC WAVEGUIDE SENSING OF MOLD FILLING AND BUBBLE FORMATION DURING LIQUID COMPOSITE MOLDING

INTRODUCTION

Although the development (1-6) of embedded acoustic waveguides (AWG) is primarily being pursued for cure monitoring of composite materials and lifetime N.D.E., these sensors can also be helpful for monitoring liquid mold filling and the gas content of resins. In work using a small transparent mold it was found that the resin flowed readily through the mold and around the glass fibers apparently without the formation of gas bubbles or voids. With an AWG immersed in the resin from one end of the mold to the other, the degree of mold filling could be estimated by measuring the reduction in the AWG transmitted signal level as the mold gradually filled with resin. However, this measurement method was not sensitive enough to detect pockets of gas trapped within the mold which were revealed when a partial vacuum was applied. It should be noted that no sounds (~3 kHz to ~80 kHz) were detected as these gas bubbles formed and reached the resin surface over a time period of a few seconds. Presumably, this was because of the viscous resin cushioning the bubbles and the sound dampening properties of the resin.

Based on previous observations of AWG signal variations during AWG resin cure monitoring, it was hypothesized that non-visible gas bubbles may be formed within the resin during the exothermic temperature rise. Consequently, in an attempt to further understand the AWG response to gas in the resin, a series of experiments was carried out. These experiments covered simultaneous AWG cure curves of resin poured normally and resin degassed before pouring; and several records versus time of the AWG signal level transmitted between immersed AWG for mineral oil and resin as normal and when saturated with nitrogen gas; resin under atmospheric air, under vacuum, and under atmospheric nitrogen; and ginger ale both saturated with carbon dioxide and flat.

The most significant results were the order of magnitude AWG signal reduction when resin was saturated with nitrogen and no gas bubbles were visible, and the order of magnitude AWG signal increase when ginger ale was saturated with carbon dioxide; and in both cases the time to return from the saturated to normal condition was about two to three hours.

Based on these experimental results, it is believed that embedded AWG will prove to be helpful for determining when liquid composite molds are filled with resin and also yield some information on whether gas bubbles are generated during mold filling.

AWG CURE MONITORING

Laboratory experiments of AWG cure monitoring of Shell 815 resin were carried out using molds of the type shown in Figure 1. The signal level transmitted through the AWG is recorded throughout the cure cycle and this signal is very sensitive to changes in the acoustic impedance (density (ρ) x acoustic wave velocity (c)) of the medium surrounding the AWG. Consequently, during the molding of a material, such as a resin, as this changes from a liquid to a highly viscous solution; to gelation (transition from a liquid state to that of a rubbery gel); to a more rigid material; to a high modulus state; the attenuation of signals within the AWG tracks these different states.

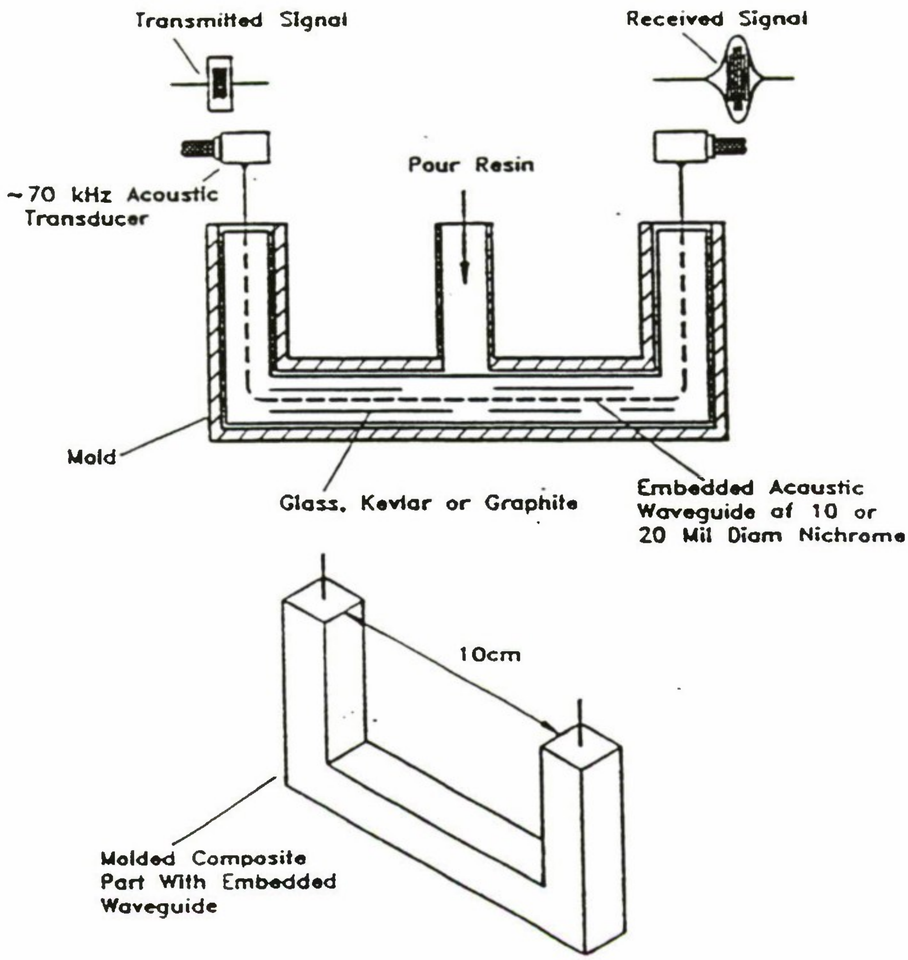


Figure 1. Acoustic waveguide cure monitoring of molded composite part

Mold filling experiments were carried out using an aluminum mold similar to that shown in Figure 1, but with a transparent polycarbonate face so that the resin flow could be observed. In addition, resin was poured into one end of the mold during filling and the central resin pouring region was eliminated.

MOLD FILLING: As resin was poured into one end of the mold with a transparent face, Figure 1, it was observed that the resin flowed readily through the mold and around the glass fibers apparently without the formation of gas bubbles or voids. It took some time, ~16 minutes, to fill this small model mold completely, as the resin was gravity fed. In addition, as the resin gradually filled the first leg of the mold, the AWG signal would gradually reduce in magnitude, then remain steady, until the bottom part of the mold filled sufficiently with resin to cover the AWG in that location. The resin flow rate was reducing at this time and vacuum application was required to pull the resin up the final leg of the mold. This vacuum application caused the release of trapped gas bubbles and these were observed for several seconds emerging from the mold lower surface and traveling to the resin surface at the final leg of the mold.

Although this type of AWG measurement of mold filling, Figure 2, is not sensitive enough to detect the presence of trapped gas bubbles, it does provide a form of measurement which could be useful for repeated use of a particular mold. With large size practical molds, several AWG would most likely be required.

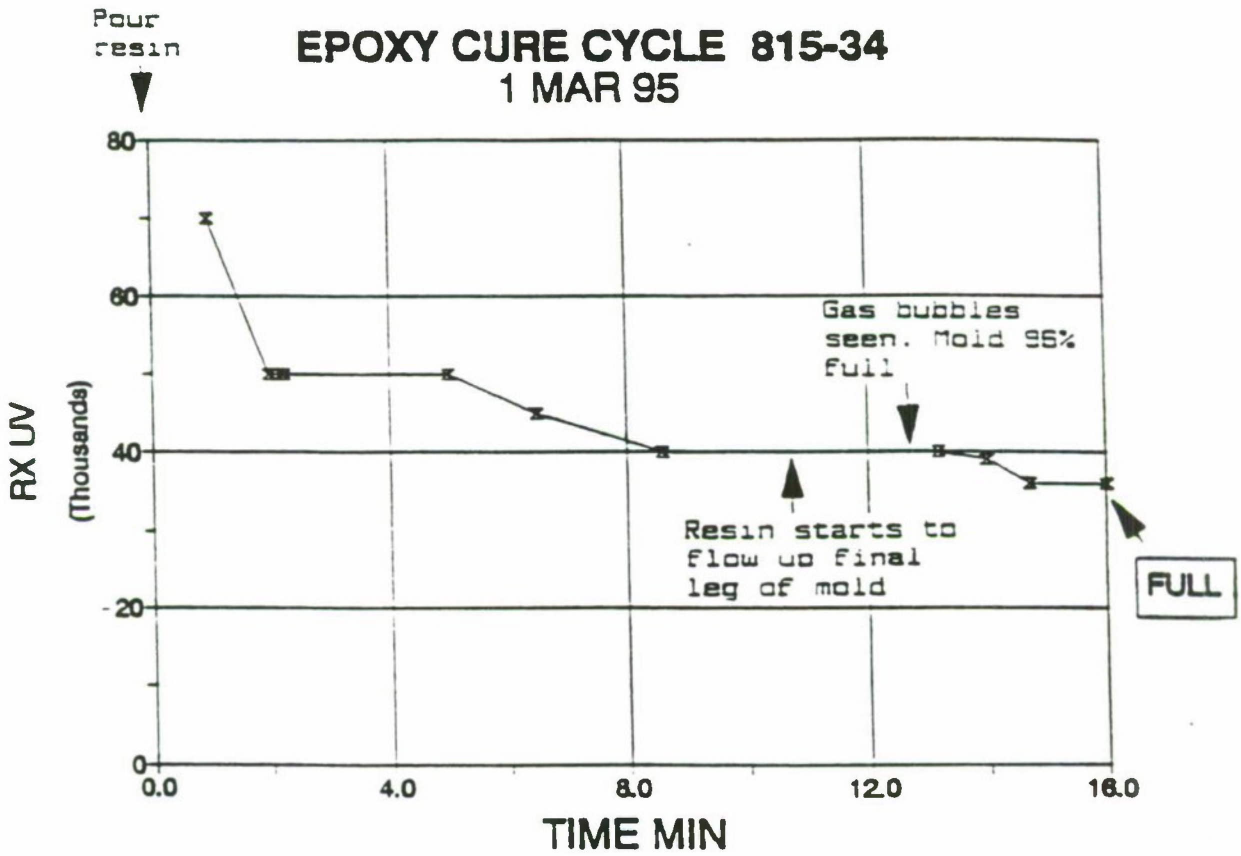


Figure 2. AWG signal levels recorded during filling of the mold with resin

GAS BUBBLES: When the gas bubbles were observed during mold filling the embedded AWG was also used in the listening mode (~3 kHz to ~80 kHz) to determine whether the generation and flow of bubbles to the resin surface caused any acoustic emissions, as is the case for less viscous liquids (7). No acoustic emissions were sensed, and it is believed this is because of the cushioning and sound dampening properties of the viscous resin.

AWG CURE MONITORING OF NORMAL AND "DEGASSED" RESIN: In an attempt to determine whether Shell 815 resin when mixed with hardener and ready for pouring into a mold contains trapped gas, simultaneous AWG cure curves were obtained using normally mixed and "degassed" resin. Both resin samples were mixed at the same time and one sample was placed under a partial (<50 Torr) vacuum for 10 minutes. The molds used for cure monitoring were aluminum containers holding about 200 cm^3 of resin each, and the simultaneous AWG cure curves are shown in Figure 3. These curves are very similar, especially in the time after resin pouring and when the viscosity is increasing, also the gelation phase time and changes. The hardening phase parts of the cure curves are also similar, with the non-degassed resin curve indicating a higher signal level and presumably a slightly higher modulus. From this experiment it can be concluded that degassing this type of resin before pouring has no obvious effect on the cure curve. For example, if the gas content of the resin samples was substantially different after pouring, then due to acoustic wave attenuation and bubble dispersion, the initial parts of the two cure curves up to gelation would be anticipated to be different. It should also be noted that at the maximum exotherm temperature rise near 88 minutes, the shape of the two cure curves is very similar. This is another indication of a similar gas content for each resin sample.

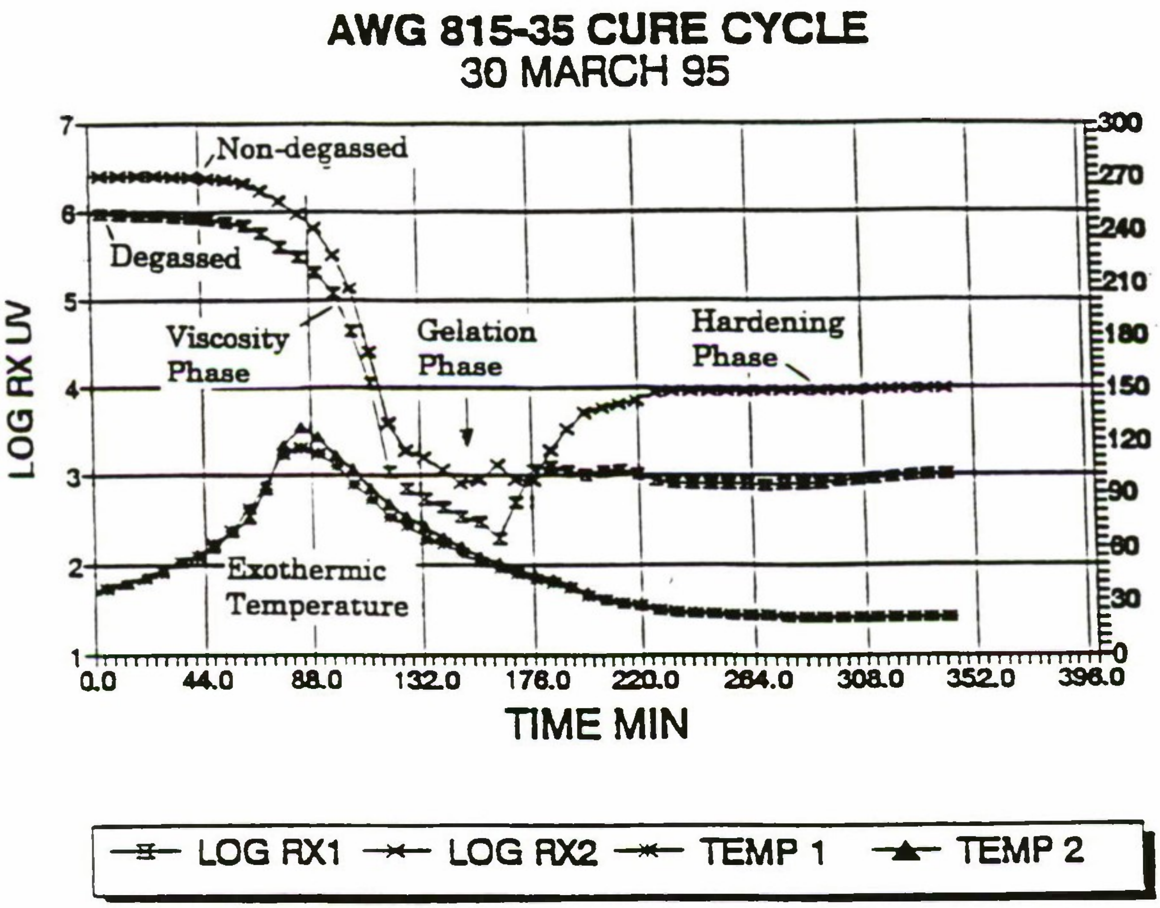


Figure 3. Simultaneous AWG cure curves for Shell 815 resin cured at room temperature using both degassed and non-degassed resin(0 to 350 minutes)

CHANGING THE GAS CONTENT OF RESIN: In order to gather information in regard to the gas content of resin, a series of experiments were carried out using the apparatus shown in Figure 4. This system allowed AWG monitoring both along an AWG as usual, and also between two AWG spaced ~2 cm. It was felt that monitoring between AWG would be more sensitive to the gas content of resin. The resin was saturated with nitrogen gas using a bubbling method first used several years ago (7) in mineral oil. Results for mineral oil and repeated in this study are shown in Figure 5. Basically, nitrogen gas is bubbled into the mineral oil via a perforated submerged pipe and the variations in the acoustic signal level transmitted between immersed AWG is recorded.

As can be seen in Figure 5 for mineral oil, after a bubbling for about 30 minutes, the AWG signal level reduces by an order of magnitude. At 60 minutes, the bubbling is turned off, no bubbles are visible, yet the AWG signal remains reduced and takes a few minutes to return to its original level. Similar results for Shell 815 resin (without hardener) are shown in Figure 6. The main differences are 20 minutes of nitrogen bubbling and over two hours to recover to the normal signal level, all without visible gas bubbles. Long recovery time would be anticipated for the more viscous resin compared with mineral oil. Actual oscillographic pictures of the signals transmitted between AWG for the saturated and non-saturated conditions for both liquids are shown in Figure 7.

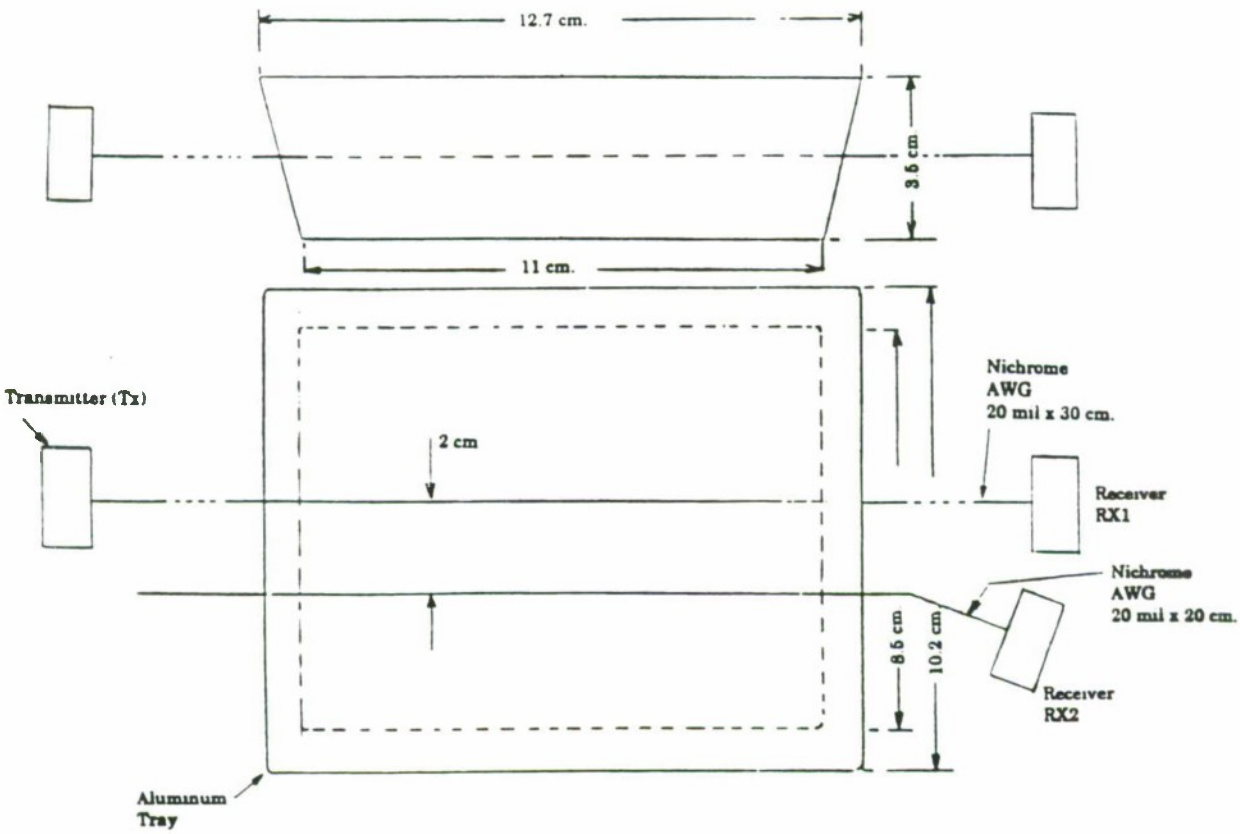


Figure 4. Arrangement for the acoustic waveguide cure monitoring of resin by measuring the signal attenuation along one waveguide and between two waveguides

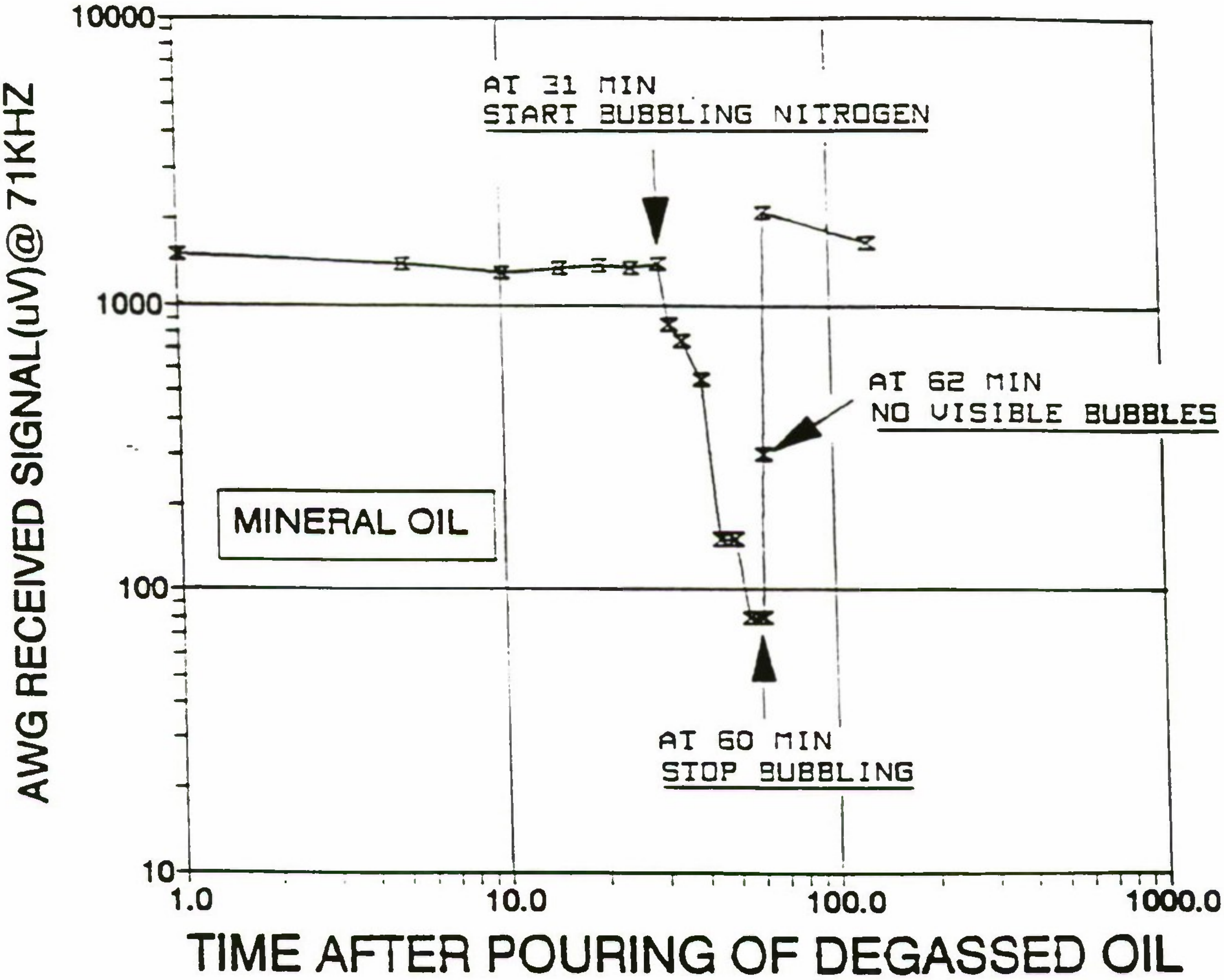


Figure 5. The influence of the gas content of mineral oil on the attenuation of ultrasound transmitted between immersed AWG

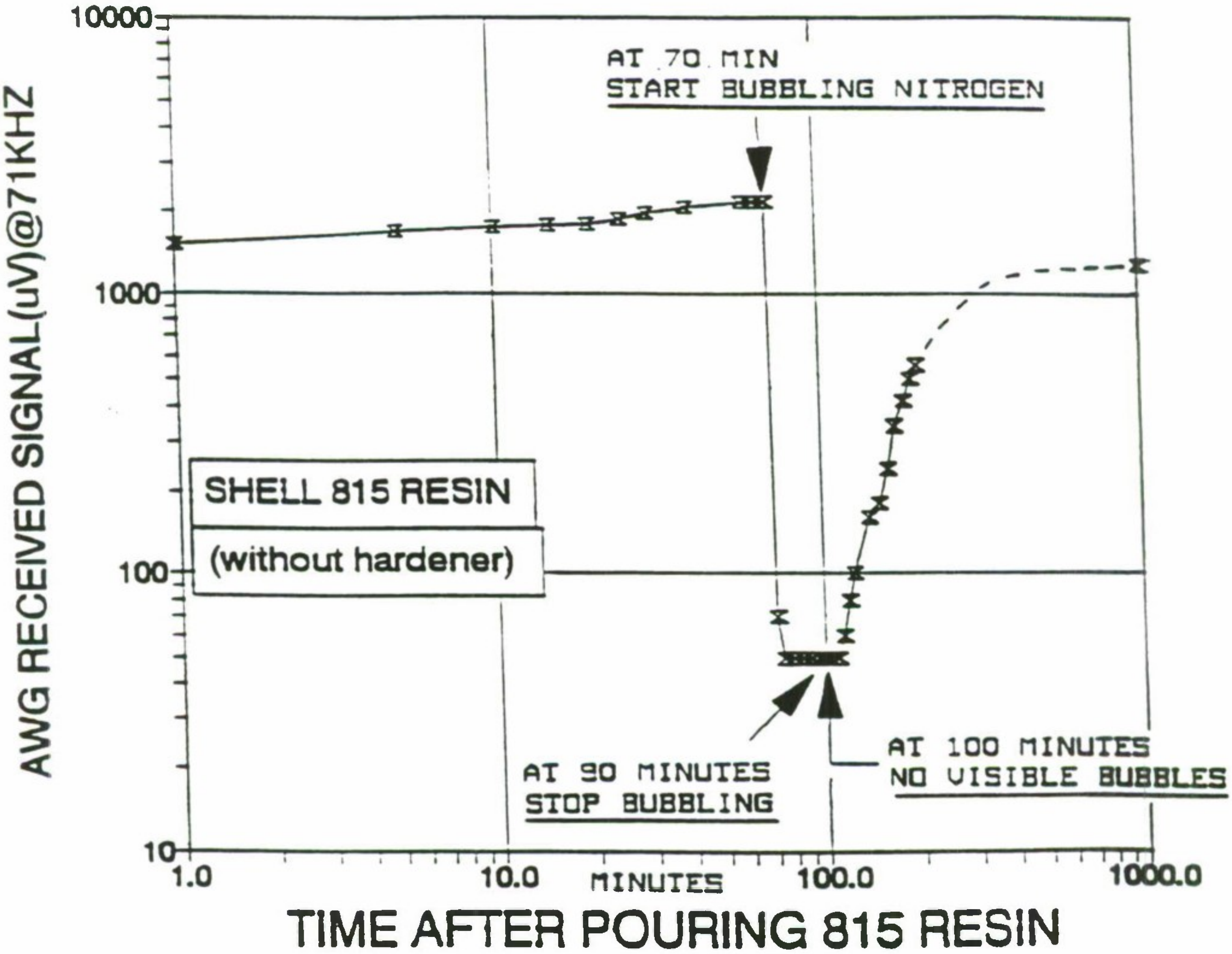
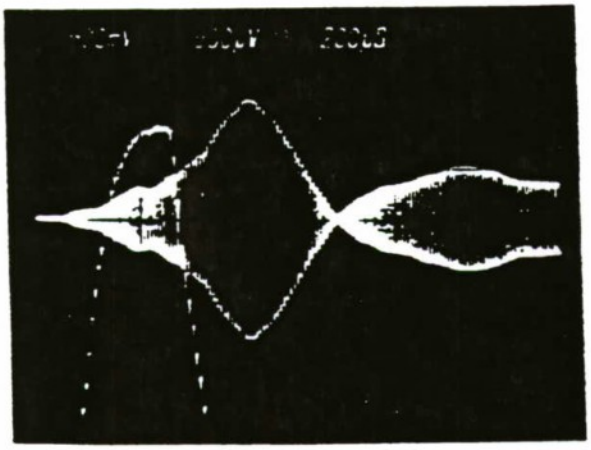
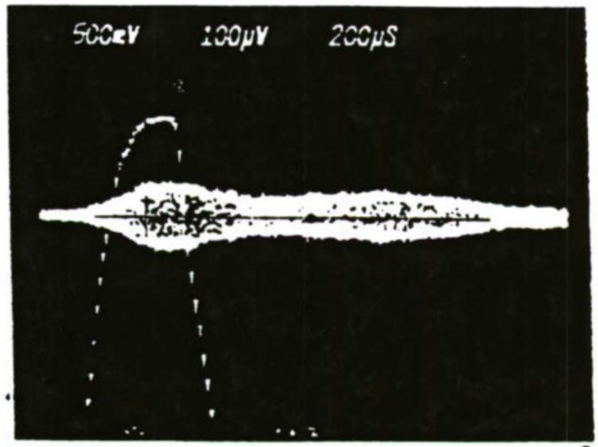


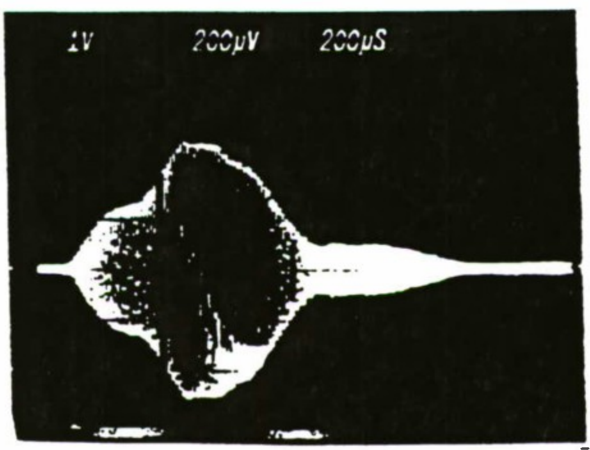
Figure 6. The influence of the gas content of Shell 815 resin on the attenuation of ultrasound transmitted between immersed AWG



AWG signal for mineral oil at 64 minutes, 25 seconds, no visible bubbles. Signal level ~1200μV and returning toward original 1500μV level.



AWG signal for mineral oil at 60 minutes, 20 seconds, no visible bubbles. Saturated condition. ~70μV.



AWG signal for Shell 815 resin at 185 minutes, no visible bubbles. Signal level 500 μV and returning toward original 1500 μV level.

AWG signal for Shell 815 resin at 100 minutes, no visible bubbles. Saturated condition, ~50 μV

Figure 7. Attenuation of signals transmitted between AWG immersed in both mineral oil and Shell 815 resin when saturated with nitrogen gas

CHANGING THE GAS CONTENT OF RESIN DURING THE CURING CYCLE: In order to determine whether signal transmission between AWG would sense the presence of gas in the resin during the cure cycle, simultaneous AWG cure curves were obtained for Shell 815 resin for signal transmission along an AWG and between AWG, Figure 8. Approximately 10 minutes after pouring of the resin the nitrogen bubbling within the resin was activated for a 10 minute time period. As can be seen in Figure 8, the gas saturation condition was sensed by the order of magnitude reduction in the acoustic signal level transmitted between AWG, but not by the signal being transmitted along one AWG. Furthermore, the reduced signal level sensed between AWG remained when the nitrogen bubbling ceased, and only returned to the apparent normal level about 40 minutes after pouring of the resin. This experimental result provides convincing evidence that the monitoring of acoustic signal levels transmitted between immersed AWG during the resin cure cycle can yield information on the gas content of the resin.

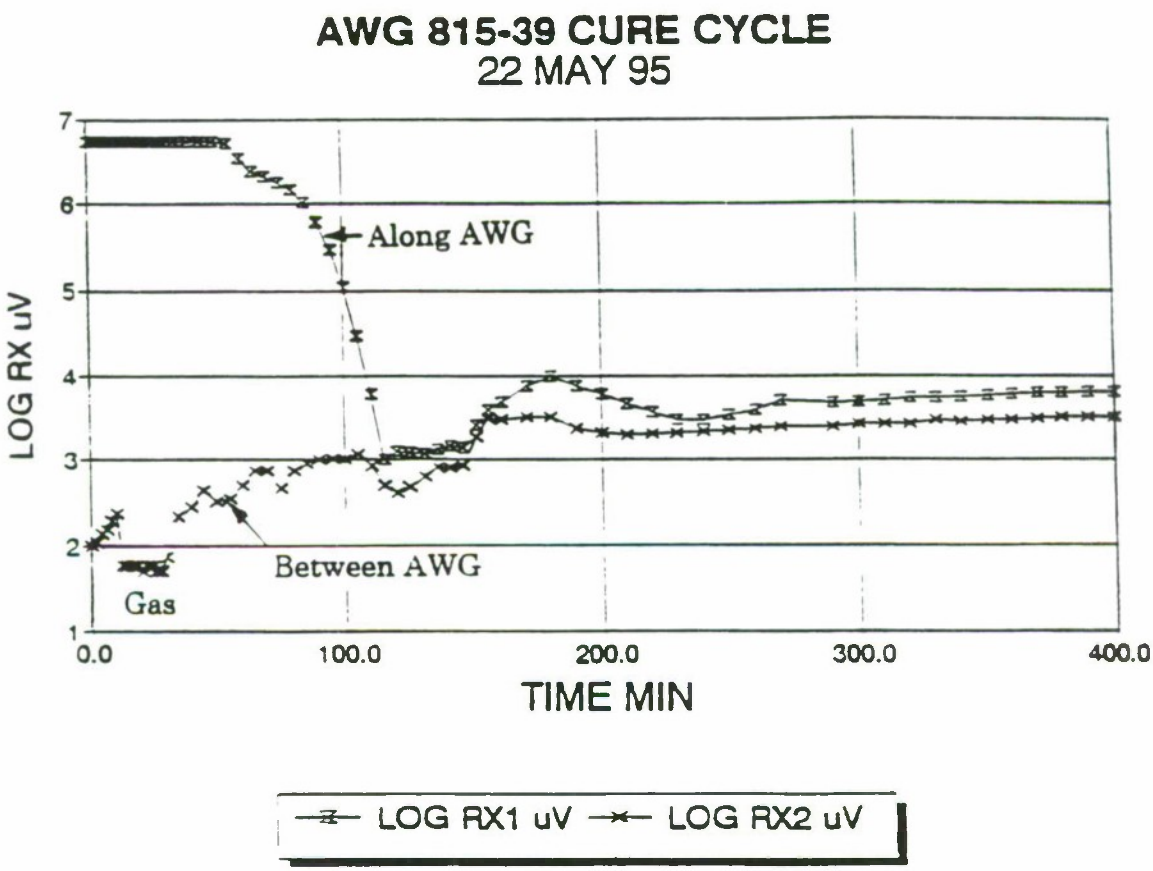


Figure 8. Embedded AWG cure curves for Shell 815 resin Specimen #39. AWG transmission along and between AWG. Gas bubbles in resin sensed by transmission between AWG

In addition, acoustic waveguide transit times (velocity) both between AWG and along an AWG were recorded during this experiment, Figure 9. As might be expected under gas saturation conditions, the wave transit time between AWG slowed up, with a change of transit time of ~90 μsec (in the resin) to ~180 μsec (in the gas). This can be roughly interpreted as 90 μsec wave travel time for 2 cm between AWG, or ~220 m/sec, which is of the order of that expected for nitrogen gas; i.e., 330 m/sec. Transmission velocities within curing resin are in the ~1000 to ~2000 m/sec range.

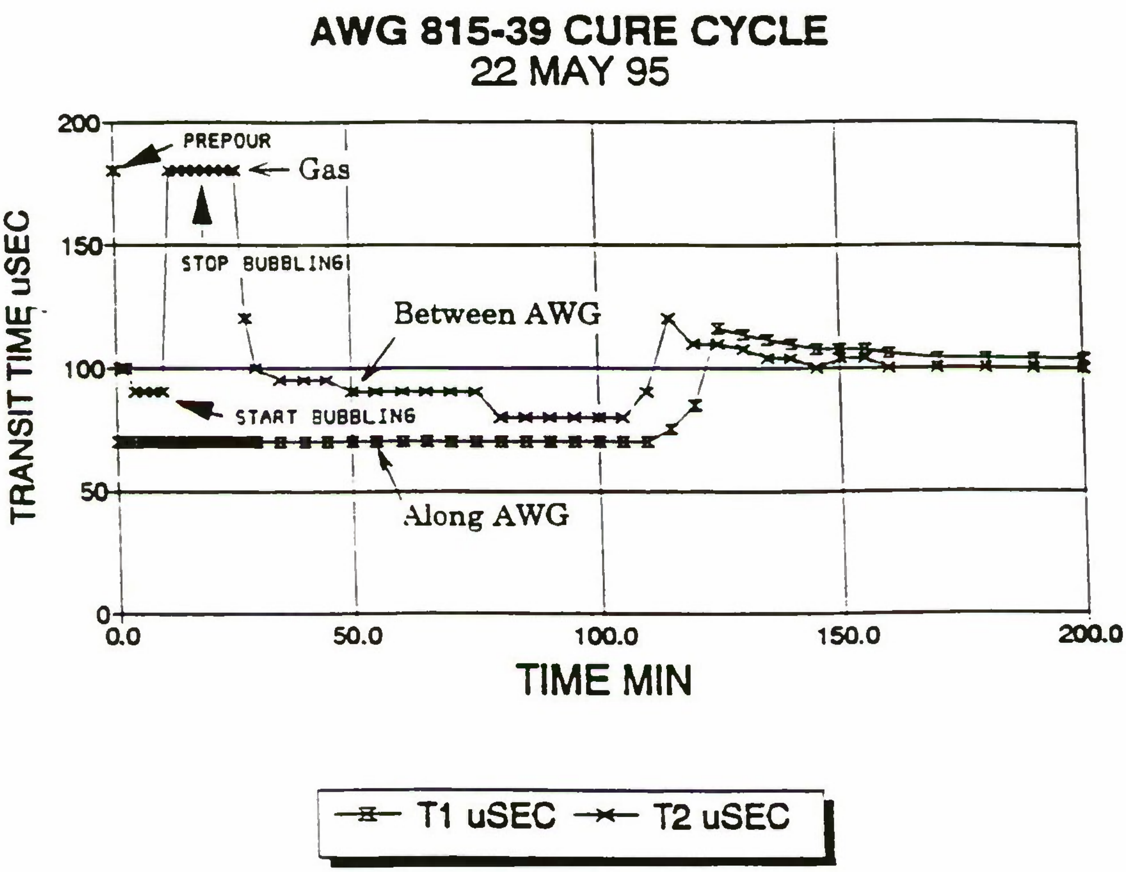


Figure 9. AWG transit times (velocity) along AWG and between AWG versus cure time for Shell 815 resin Specimen #39. Transit time changes between AWG indicate resin gas content

LONG TERM AWG MONITORING OF RESIN: It was observed that if the acoustic signal transmission between immersed AWG was monitored daily for resin (without hardener), different cyclical variations in the signal level occurred for resin in atmospheric air and in atmospheric pressure nitrogen, Figure 10. As can be seen in Figure 10, the daily signal fluctuation for resin in a nitrogen atmosphere is from ~300 µV to ~450 µV; or about 50%. Whereas, for air, the variation is from ~320 µV to ~350 µV, or about 10%. These results demonstrate the AWG system sensitivity and probably indicate cyclical resin gas absorption changes.

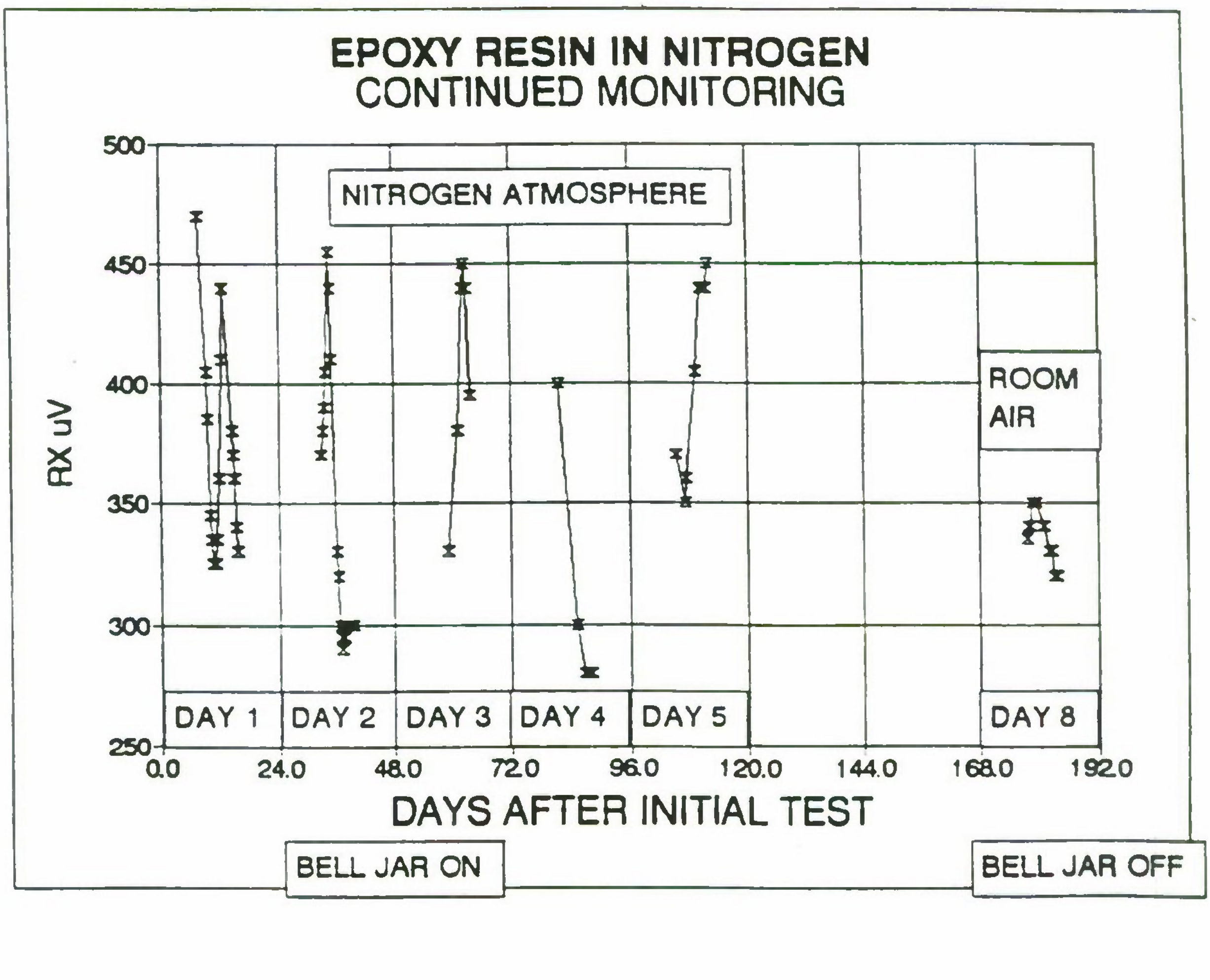


Figure 10. Daily cyclical variations of acoustic signal level transmitted between AWG immersed in Shell 815 resin (without hardener) for resin in atmospheric nitrogen and atmospheric air

AWG MONITORING OF RESIN UNDER VACUUM: In order to learn more about the phenomena being measured, the resin (without hardener) was placed under vacuum while the signal transmission between AWG was monitored, and then the bell jar used for evacuation was filled to atmospheric pressure with nitrogen, Figure 11. As was anticipated, when the resin was under vacuum (gas withdrawn from resin) the AWG signal increased in magnitude, and when atmospheric nitrogen covered the resin, the AWG signal reduced (gas introduced within the resin). However, these changes were general, and not precise, and obviously the exact phenomena occurring are not fully understood. Nevertheless, these data do give credibility to AWG monitoring of the resin gas content.

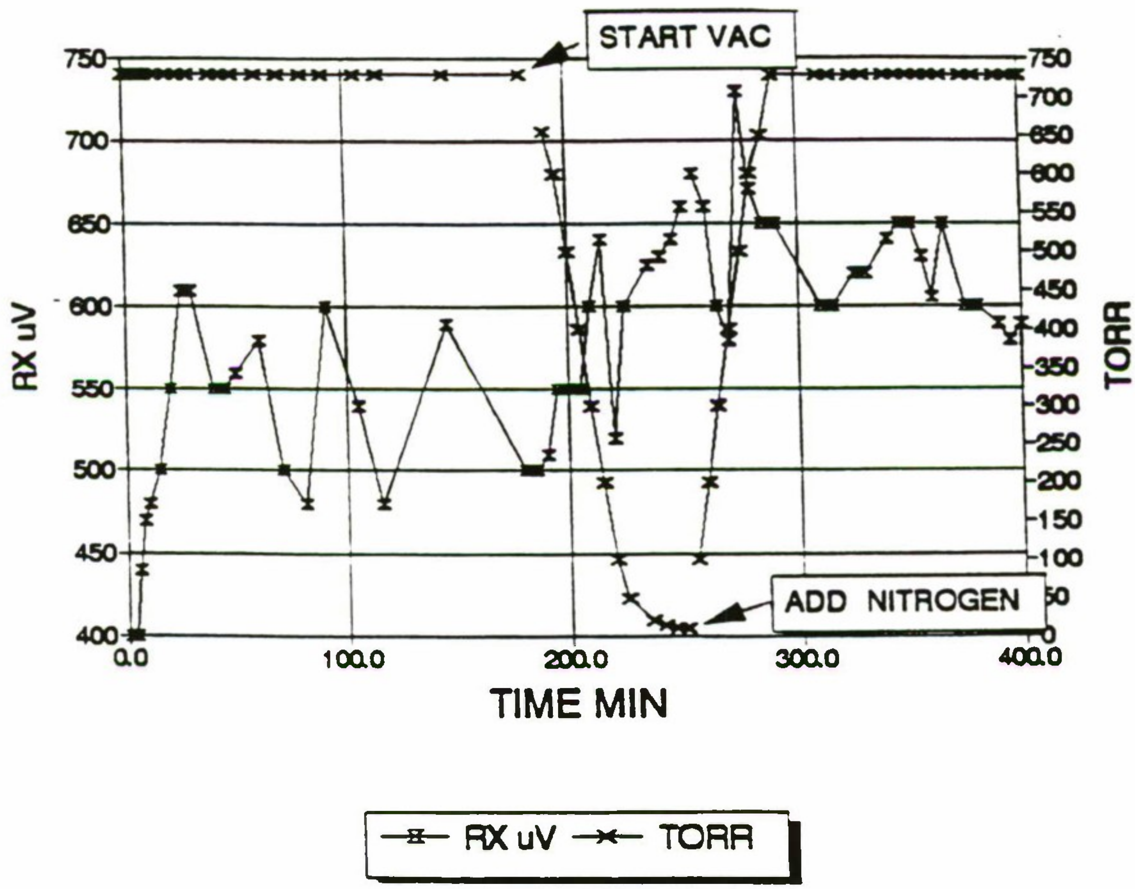


Figure 11. Variation in acoustic signal level transmitted between AWG immersed in resin for resin in atmospheric air, vacuum and atmospheric nitrogen

AWG MONITORING OF GINGER ALE FIZZ: It was reasoned that if the AWG system was sensitive to the gas content of resin and mineral oil, then it should be able to sense the carbon dioxide content of common liquids, such as, ginger ale. AWG monitoring results for the gas content, or fizz, of ginger ale after pouring into an open container in room temperature air are shown in Figure 12. Clearly, the AWG system monitors the gas content of ginger ale from the maximum fizz, to minimum fizz, to flat condition, in two to three hours. In addition, the sound pressure waves from surface bubbles bursting raises the signal level transmitted between AWG as indicated. A similarity with the resin and mineral oil results is the order of magnitude signal level change from the gas saturation to flat condition. A striking difference is the maximum signal transmission for gas saturation, and minimum signal transmission for the flat condition, the reverse of the resin and oil data. This may be due to the special ~60 kHz acoustic properties of carbon dioxide.

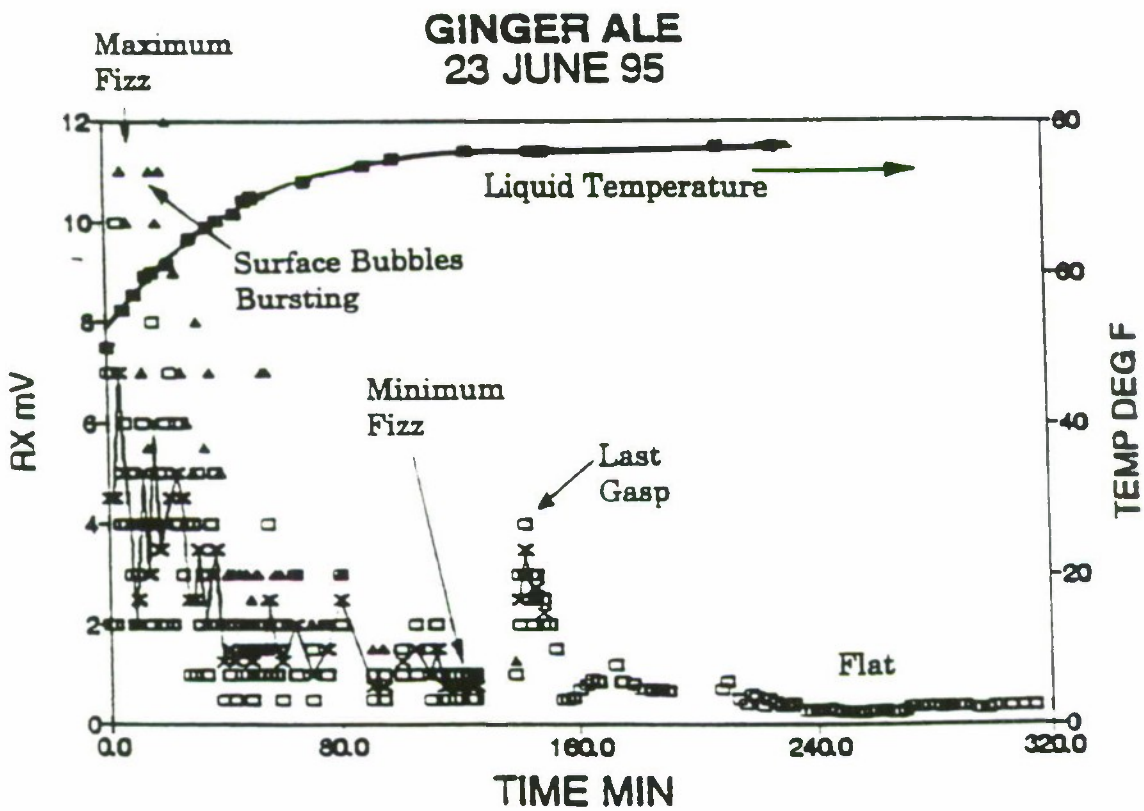


Figure 12. Signal level transmitted between AWG immersed in ginger ale versus time after pouring cold liquid

CONCLUSIONS AND DISCUSSION

It has been demonstrated that monitoring the signal transmitted along an embedded AWG can be helpful for giving an indication of the degree of liquid mold filling, but cannot sense the presence of trapped gas bubbles. On the other hand, monitoring the signal transmission between embedded and parallel AWG can yield information on the gas content of curing resin. Clearly, for the most information during resin curing, so that processing may be controlled, it would be best to monitor both the signal transmission variation along and between embedded AWG.

This study has also yielded new information, not yet fully understood, which generally indicates an order of magnitude AWG signal attenuation when liquids are saturated with gas, but no bubbles are visible to the naked eye. In addition, the time for the liquid to return to the non-saturated condition is related to the liquid viscosity. It is also believed that degassing of resin before pouring may not be helpful, and that gas or void production within curing resin is primarily related to processing.

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